

École polytechnique de Louvain

Enhancement of the optical properties of silicon devices through strain engineering

Development of a lab on-chip devoted to biaxial loading of a silicon thin film specimen

Author: **Raphaël MARICHAL**

Supervisors: **Jean-Pierre RASKIN, Thomas PARDOEN, Denis FLANDRE**

Readers: **Marie-Stéphane COLLA, Nicolas ROISIN**

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List of acconyms

BHF	Buffered hydrofluoric acid
BOX	Burried oxide layer
CA	connector/actuator interface neighbourhood
CC	Rounded region between consecutive connectors
CMOS	Complementary Metal Oxide Semiconductor
CPD	Critical point drying
DFT	Density functional theory
DI	Deionized water
FE(A)	Finite element (analysis)
HF	Fluorhydric acid
ICP	Inductively coupled plasma
LPCVD	Low pressure chemical vapor deposition
PECVD	Plasma enhanced chemical vapor deposition
PEB	Post-exposure baking
RIE	Reactive ion etching
SEM	Scanning electron microscope
SOI	Silicon-on-insulator
TEM	Transmission electron microscope
WINFAB	Wallonia Infrastructure for Nano-FABrication

Abstract

The trend towards miniaturization of electronic devices requires to evaluate the change of physical properties of materials at nanoscale. The present work aims at developing an on-chip laboratory devoted to put a silicon thin film specimen under biaxial loading, and to investigate the change of its materials properties. Finite elements simulations are used to derive convenient specimen designs, and to extract a set of dimensions allowing to sweep a range of strains from 0.001 to 0.04. Labs on-chip, based on SOI technology, are then fabricated in cleanroom.

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Chapter 1

Context

For several decades, microelectronics has taken a tremendous importance in the actual word, and has strongly modified our way of life. The on-going trend of the society aiming at growing the part of automation of human tasks calls for the development of electronic-based devices even more efficient and reliable, which requires research and development to investigate new routes, still unexplored, in electronics, physics, chemistry and material science. In addition to this wish of increasing efficiency, firms linked to electronic field have the purpose to constantly decrease the size of their devices : developing a smartphone or a laptop as thin as possible, offering some sensors as small as possible, or in a more general way decreasing the size occupied by electronics in the commercialised products. Several breakthroughs, due to the tremendous researches in the field of nanotechnology, permitted any incredible progress in this quest of down-scaling devices development, leading to the creation of small size, efficient and reliable devices. Despite this trend permits to significant enhancement in several domains, some of them still suffer from a lack of notable improvement for specific applications, and that is why scientist community mobilizes efforts to fill this gap, daring sometimes getting off the beaten tracks.

Existing routes to increase the electronic device efficiency are quite various. One of these routes, not the most investigated one by the past, recently turns out to present some attractive advantages : the *strain engineering* route. Its working principle is quite simple : a stress is applied to a part of the device, modifying the lattice of a functional layer. Following the crystallographic formalism, a change in the direct lattice induces a change in the reciprocal one, responsible of electronic properties of materials. Consequently, the primitive cell of this reciprocal lattice, called Brioullin zone, is modified, and the associated band structure defined along the segments existing between its connecting points too. The effective mass of charge carrier being linked to the band curvature, modifying this band structure has an interesting impact on this parameter, changing the charge carrier mobility and in the electric conductivity expressions.

This *strain engineering* route has being investigated for instance in CMOS technology[1] and turns out to lead to higher charge carrier mobility and faster switching time than conventional unstrained devices [2]. Since early 80's, it has also emerged in the field of opto-electronics and photonics, with PIN diodes[3] for instance. Indeed, in opto-electronic devices, the modification of the band structure by applying strain permits to

tune the bandgap energy, modifying the absorption spectrum and influencing on the cut-off frequency. So strain engineering could be a way to solve for instance the lack of photodetectors efficient in the spectral range or in the short-wavelength infrared, which could have a great utility in self-driven vehicle industry for instance [4]. In the same way, it has been shown that strain-engineering based PIN photodiodes, composed of *Ge/GeSn/Ge* heterojunctions, where the *GeSn* active layer is fully strained, presents stronger response than optical devices with single unstrained active *Ge* layer [5]. However, the number of recent papers dedicated to strained engineered remains is quite low. Performing deeper investigations in this way can so permits to obtain more efficient devices and to make up for a lack of particular applications, like near-infrared photodectector for instance.

Nowadays, due to the ever continuing drive towards down-scaling, several attempts are performed to build such electronic devices with size beyond actual limits in term of size to reach an ultra high scale of integration. At nanoscale, its possible to reduce the dimensionallity of the material in one direction (to get 2D objects, like thin films), two directions (to get 1D objects, like nanowires) or even three directions (to get 0D objects, like quantum dots). Building structures at this scale can be quite challenging, but thanks to the advent of micro electromechanical devices, commonly called *MEMS*, some micro and nanofabrication techniques were developed to build structures at such scales.

However, when trying to combine the quest of miniaturization and the efficiency enhancement for instance through strain engineering as presented, scientist community faces an intricate problem : material properties become size dependent at nanoscale, and can even strongly differ from the bulk ones. Indeed, when the dimensionallity of the system decreases, the surface-to-volume ratio strongly increases. Consequently, surface driven processes play a more important role as physical properties of the material is concerned. Having fundamental understanding of the physical processes involved in this size dependence of material property is essential to design an efficient and reliable device.

Since early 60's, one of the mostly used material in the field of microelectronics is silicon. Its various attractive properties made it considered by scientific community as the foundation stone of electronic systems present in nearly every domain of everyday life : communications, energy, transports, data processing, health cares... But why is silicon so interesting ? The answer is quite straightforward : its the most abundant materials on earth's crust (so its price is low) and its has the property to be a semiconductor. Moreover, its electronic properties can be quite easily tuned to fulfill specific roles, broadening its potential uses. The use of silicon is widely spread in a huge majority of entities underlying "electronics": to name a few of them, in which silicon has played a important role, one finds state-of-the art transistors, micro (even nano) electromechanical devices, opto-electronic and opto-mechanical structures or phononic crystals [6].

As explained above, the development of efficient silicon devices, while decreasing its size to gain in integration, requires to anticipate its behaviour at nanoscale. Performing investigations to understand the physical behaviour of silicon objects at this scale is so primordial, because it constitutes the first stage of the device development. This field has

already been quite broadly investigated and quite strong changes in physical properties were pointed out [7],[8]. However, some methods used to perform these investigations owned some drawbacks or limitations, which explains why scientific community struggle to develop new testing method to extract those informations. Extending investigations in this domain could lead to tremendous breakthroughs in some applications in silicon technology, like the ones presented before.

This master thesis has the aim to continue this investigation, more specifically concerning the change of mechanical, optical and electronic properties of silicon at microscale through strain engineering, by proposing a direct characterisation by an on-chip investigation. The concept of lab on-chip was partially developed and investigated by prof. Raskin's research group from UCLouvain and the associated fabrication process has already been investigated. Such devices presents strong attractive features, by allowing a direct on-chip accurate investigation of several physical properties of materials, allowing statistical analysis. The present investigation is devoted to extend the work performed by UCLouvain research team, by developing a scientific methodology to apply the on-chip characterisation to free-standing single crystal silicon thin films undergoing biaxial tensile stress. Indeed, each loading mode (uniaxial, biaxial, triaxial...) has a particular influence on silicon physical properties, because it modifies the crystal symmetry in a particular way. The choice of biaxial loading is justified by several reasons. Here is a non exhausting list of reasons making biaxial loading appealing for investigation :

- Lower strains are needed in biaxial loading than in uniaxial solicitation to reach the fracture strain
- Biaxial loading induces shear effect, and causes a particular degeneracy lift in silicon band structure which can be useful to obtain particular band-to-band transitions specific to some optoelectronics applications.
- It has been found that biaxial strained silicon layer exhibits enhanced charge carrier mobility.
- Uniaxial strength alone is insufficient for predicting fractures in complex stress fields : an both uniaxial and biaxial fracture strength are often needed, and available information about biaxial strength is quite weak.
- It constitutes a complementary way to uniaxial tensile test to investigate the change of materials property at nanoscale. Indeed, crystal symmetry is modified in a different way under biaxial strain than uniaxial strain.
- It is a loading mode which has not been thoroughly investigated through on chip investigation. In the case of success, this on-chip characterisation could add to the other mode investigated by UCLouvain research group, like uniaxial loading or shear loading.

The field investigated here will thus be more oriented towards materials science to deduce material properties, aiming to be used afterwards as basis to design particular strained engineering based electronic devices.

The present manuscript is divided in the following way : *Chapter 2* gives the physical background needed to understand the concept of strain engineering, in addition to the review of existing biaxial tensile testing structures, to show how advantageous and powerful is the lab on-chip one. *Chapter 3* is dedicated to the design of convenient structures for biaxial loading. Fabrication process in *WINFAB* facilities is then exposed in *Chapter 4*, followed by a brief characterisation section in *Chapter 5*.

Chapter 2

State of the art

One has just to read the tremendously long title of this master thesis to notice that several branches of physics will be involved here. To clearly present the concept of *strain engineering* for semiconductors, a reminder concerning some material properties has to be given. The first part of the state of the art briefly present crystallographic, opto-electronic and mechanical properties of single silicon. This background permits to understand, in the second part, the effect of strain on opto-electronic properties and to present the principles on which strain engineering is founded. Thirdly, a review of existing structures devoted to biaxial tensile testing is given, by pointing out the pros and cons of each technique. The concept of lab on-chip is then introduced, which has already permitted to characterize various physical properties of nano-objects like nanowire and nanoribbons. Those structures presents numerous advantages, justifying its interest by a part of scientist community. This chapter ends by the tool used to characterize these structures once fabricated.

2.1 General properties of single crystal silicon

2.1.1 Crystallographic properties

Silicon is the 14th element of the periodic table. It crystallizes in a diamond structure, consisting in two interpenetrating face-centered cubic (FCC) lattices, shifted by a vector $(\frac{1}{4}, \frac{1}{4}, \frac{1}{4}) a_0$ where a_0 is the lattice parameter and has a value of 0,357 nm for silicon. All the atoms in the unit cell are sp^3 hybridized. A schema of the unit cell of silicon is given in [Figure 2.1](#). Diamond structure owns 48 symmetry elements.

One has now to introduce the notion of reciprocal space. This space is obtained by taking the Fourier transform of the direct lattice. Whereas the norm of vectors in real space can be associated to real distances between atoms, the norm of reciprocal space vectors can be associated to spacial frequencies. In such space, basis vectors are defined as¹

$$(a^*, b^*, c^*) = \frac{2\pi}{a_0} (b \times c, c \times a, a \times b) \quad (2.1)$$

¹ a, b and c refers to the unit vectors of the unit cell

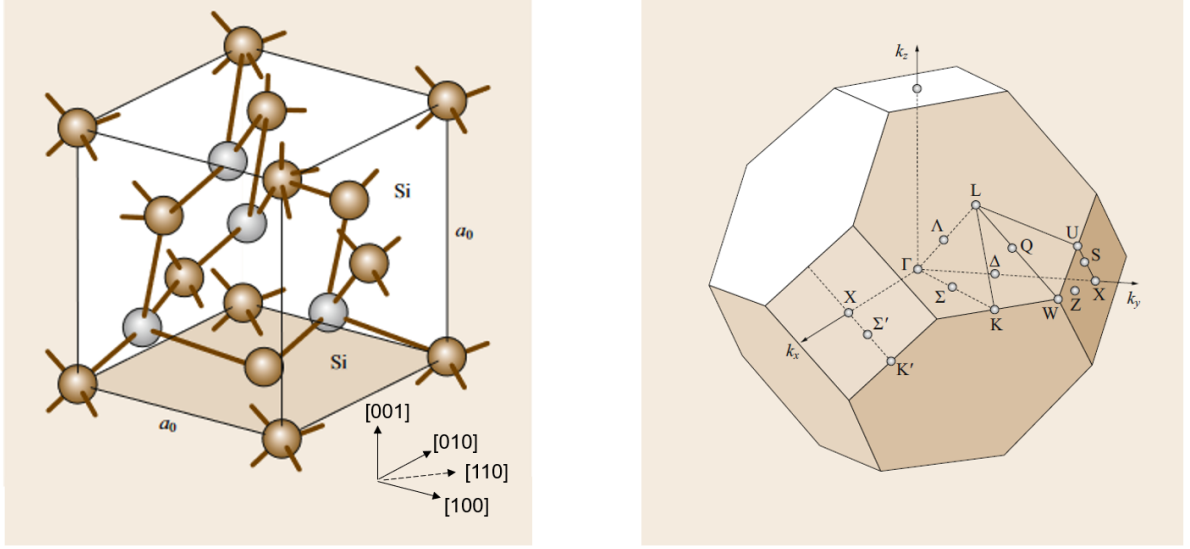


Figure 2.1: (left) : Unit cell of silicon. Atoms of the two interpenetrating lattices are represented in red and white. Lattice parameter a_0 and some crystallographic directions ([100], [010], [001] and [110]) are indicated. (Right) : First Brillouin zone of FCC crystals. Reference points are indicated. [9]

The primitive cell of the reciprocal lattice is called the first *Brillouin zone*. It is centered at the origin of the reciprocal lattice and corresponds to the volume limited by the Bragg planes existing between its center and the neighbouring positions in the k -space. This zone, analogous to Wigner-Seitz cell in the real space, has the particularity to contain all the informations about the electronic structure of the material. The symmetry operations present in the direct lattice determine the Bravais lattice of the first Brillouin zone : the unit cell of silicon being FCC, the first Brillouin zone is also FCC. Some high symmetry points present in the first Brillouin zone are used to define the band structure : for silicon, those points are L , Γ , Δ , X , U , K and Σ . Γ constitutes the center of the Brillouin and is so located at $(0,0,0)$. Points K , L , X and U are respectively located at $\frac{2\pi}{a_0}(\frac{3}{4}, \frac{3}{4}, 0)$, $\frac{2\pi}{a_0}(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ and $\frac{2\pi}{a_0}(1, 0, 0)$.

2.1.2 Electronic properties

Band structure theory is a crucial point to address if one has to describe the optical and opto-electronic properties of a semiconductor, directly linked to the electrons behaviour near the bandgap. This theory is only an approximation, which considers a perfect crystalline system of infinite dimensions. Surface and interfacial effects are so not represented in this theory, it has to be kept in mind when working with low dimensionnality objects like thin films.

Definition

Energy band theory is developed in the framework of quantum mechanics, and refers to the fact that electrons in solids own an energy comprised in well defined ranges. The allowed energy levels are called the **energy bands**, and the other energy states, forbidden for electrons, are called **energy bandgaps**. For an infinite crystal, these energy bands are continuous, and are represented by expressing the energy E of electrons for each k vector included in a segment joining two reference points of the Brillouin zone.

The band structure and associated features depends on the Brillouin zone segments. This dependence has the consequence to make band structure strongly linked to symmetry operations of the material crystalline structure. It can be explained by the fact that the electrons wavefunctions are restricted by the periodicity of the lattice. Following Bloch theorem, electronic wavefunctions can be expressed as a product of plane wave of wavevector k and a periodic function $u(r)$ having the same periodicity than the direct crystal lattice. if R is a linear combination of multiple direct lattice basis vector, electronic wavefunctions can be expressed as

$$\psi_k(r) = e^{ik \cdot r} u(r) \quad \text{with} \quad u_k(r) = u_k(r + R) \quad (2.2)$$

By writting the electronic wavefunction in this form in Schrödinger equation, the electronic states allowed for the system can be derived. The Hamiltonian in this equation is composed of a kinetic term and a mean effective potential $V(r)$, representing the potential existing between core and electrons and electron-electron interactions. The crystal being a periodic arrangement of atoms, the mean effective potential $V(r)$ has the lattice periodicity. Schrödinger equation can thus be expressed as :

$$\hat{H}\psi_k(r) = \left[-\frac{1}{2}\nabla^2 + \hat{V}(r) \right] = \epsilon_k \psi_k(r) \quad (2.3)$$

The obtained eigen energies have three properties :

- being a **continuous** function of k
- being **even** : $\epsilon_k = \epsilon_{-k}$
- Having the **reciprocal space periodicity** : $\epsilon_k = \epsilon_{k+K}$

With K a multiple of the reciprocal lattice basis vector. The last property allows thus to bring any point out the Brillouin zone within it. This existence of a one-to-one relation between eigen energies ϵ_k and ϵ_{k+K} permits to rank these eigen values in an increasing order an associated a index n to each for the identification. The solving of Schrödinger equation is so performed along the segment linking high symmetry point of the first Brillouin zone and band for each index is deduced.

Band structure calculation

Several techniques can be used to compute this band structure. A usual analytical technique used to treat materials with a low orbital overlap (like silicon) is the **tight-binding model**. This model is a one-electron model which computes the bandstructure

by considering that the electronic wavefunction of the system is the superposition of electronic wave functions of isolated atoms. This simple model considers interactions only between first order nearest neighbours atoms, leading to a limited accuracy. An example of tight binding model applied to silicon is present in [10].

Another convenient technique for determination of electronic (and optoelectronic) properties of silicon is the **ab-initio calculation**, based on the density functional theory. In this type of investigation, one uses electronic density ρ as variable instead of wavefunction. The main principle of ab-initio investigation is to consider that only valence electrons participate to the binding, core electrons are "frozen". Valence electrons move in a so-called pseudopotential, imitating real potential created by core electrons and crystal ions. The great advantage of this technique is to have access through short time calculations to positions of relaxed atoms in the unit cell and to deduce the bandstructure. The equations used in the framework of density functional theory computation is the Kohn-Sham equation :

$$E[\rho] = T_s[\rho] + \int v_{ext}(r)\rho(r)dr + E_H[\rho] + E_{xc}[\rho] \quad (2.4)$$

Where T_s is the Kohn-Sham kinetic energy, v_{ext} the external potential acting on the system (including ion-electron interaction), E_H the Coulomb energy and E_{xc} the exchange-correlation energy (a corrective term taking into account the Pauli exclusion principle and how much the movement of an electron is influenced by the other electrons in the system). This equation is solved in a self-consistent way, by using as argument some pseudopotentials : those ones contains the basis wavefunctions and the number of core electrons considered (indeed, a clear distinction between valence and core electron is quite tricky to do in some cases). Even if bandwidth and band dispection are quite well representing by using DFT, bandgaps are often underestimated, that is why the use of the GW approximation for semiconductors. This leads to a theoretical bandgap value quite close to the real one in most cases.

Band structure of silicon, degeneracy and band filling

Band structure of silicon (focused near the bandgap region) is represented in **Figure 2.2**. Like for other semiconductors, a bandgap is present between the valence and conduction band. In the case of silicon, this bandgap as a value around 1.14 eV. Silicon has the particularity to have the top of valence band present in a direction (Γ in the $k - space$) different from the one of the bottom of conduction band (on the Δ symmetry line ($\Gamma \rightarrow X$), near X). Silicon is so called an *indirect bangap* semiconductor, in opposition to *direct bangap* semiconductor, like *GaAs*, for which top of valence band and bottom of conduction band are associated to the same direction.

For the following, the concept of effective mass of a charge carrier m^* will be useful. It permits to express second Newton law for a particle of the system, by considering in the left term only the external applied force. This effective mass term includes the effect of the lattice on the particle, and depends thus on the lattice properties. For a particular band, the mass effective is inversely proportional to the band curvature : higher the curvature

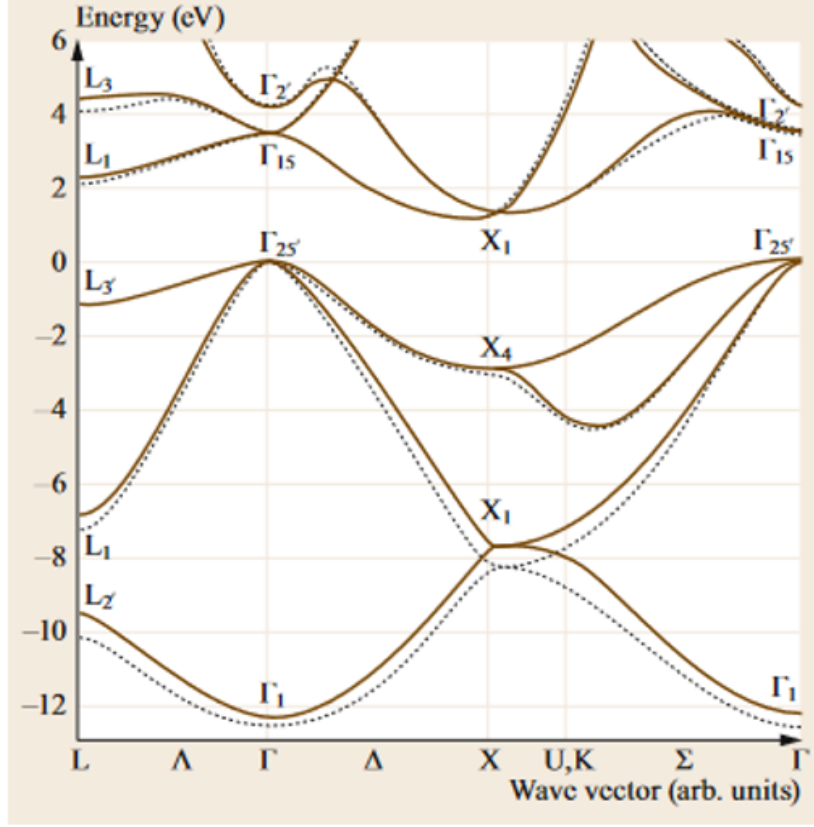


Figure 2.2: Band structure of silicon [11]

is, lower is the effective mass.

The symmetry existing in the lattice induces some energy band degeneracy. The highest order degeneracy for a band equals the number of coordinates transformed by the symmetry operations. Silicon valence band having a contribution of p-atomic orbitals, it results in a threefold degeneracy at the Γ point : the k_x , k_y and k_z directions in the Brillouin zone are equivalent so the energy diagrams along these directions are the same. In the band structure diagram, one can identify two bands : a heavy-hole band and a light-hole band. These concept of heavy and light charge carrier refers to the effective mass, for which absolute value decreases when the band curvature increases. The bottom of the conduction bands is located on the $[\Gamma - X]$ segment (about $0.85X$) and is constituted of six equivalent valleys Δ_6 , located along the $\langle 100 \rangle$ directions. In opposition to the degeneracy existing due to the central position of the Γ point in the Brillouin zone (Γ), the energy equivalence of those Δ_6 valleys constitutes another kind of degeneracy : the *star degeneracy*, where energies are the same at different k-points which can become equivalent through symmetry operations (rotation, inversions...).

According Fermi-Dirac distribution, electrons fill bands successively from deepest one to the one crossing or below the Fermi level. Fermi level constitutes the maximum energy state in which the electron can be at 0 Kelvin. For intrinsic semiconductor, this level is located in the middle of the bandgap. In the case of doped semiconductor, this bandgap

is situated between the donor level and the conduction band (in the case of n-doping) or between the acceptor level and the valence band (p-doping). Without any energetic excitation (temperature increase, irradiation...), silicon as other semiconductors behaves like insulators : valence band is so full and conduction band empty.

Optoelectronic properties

Optoelectronic devices are based on electronic transition between valence and conduction band involving the absorption/emission of an electromagnetic wave. The size and the nature (direct/indirect) of the bandgap determines the particular process occurring in photon absorption or emission. For indirect semi-conductors like silicon, band-to-band transitions implies a change in both energy and momentum, whereas for direct transitions momentum is not modified. Photons being not able to change the momentum of an electron, a two step process is required for indirect bandgap transitions, involving a phonon and a photon. Light emission and absorption mechanisms for silicon are represented in Figure 2.3.

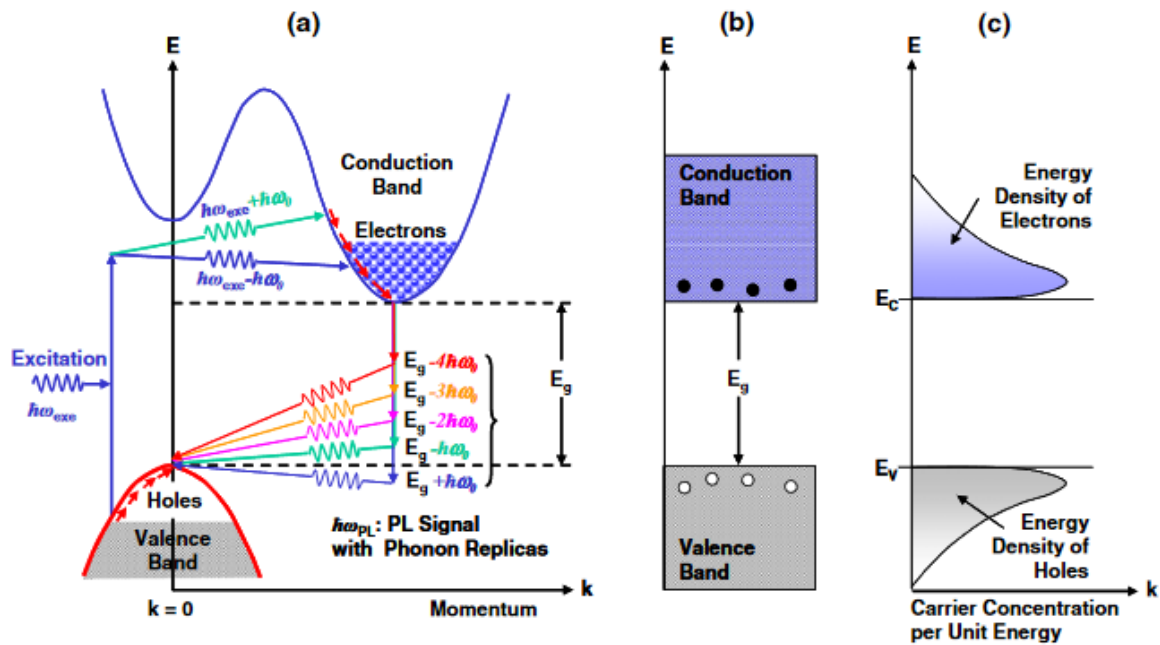


Figure 2.3: Photoemission mechanism for silicon (and other indirect semiconductors). (a) radiative recombination process. (b) energy band diagram. (c) density distribution of electrons in conduction band and holes in valence band [12]

Light absorption consists in the excitation of an electron from the valence band to the conduction band by absorbing a photon, creating a hole in the valence band. This transition from fundamental to excited state is performed by simultaneously absorbing a photon and absorbing (blue array) or emitting (green array) a phonon in the case of indirect bandgap semiconductors. Figure 2.4 shows silicon absorption coefficient as a

function of photon energy (on left). The absorption begins around the indirect bandgap energy (1.1 eV). Two peaks at higher energies are present (E_1 and E_2), stemming from the parallelism existing between valence and conduction band [13]². Photodetectors are a kind of optoelectronic devices based on light absorption.

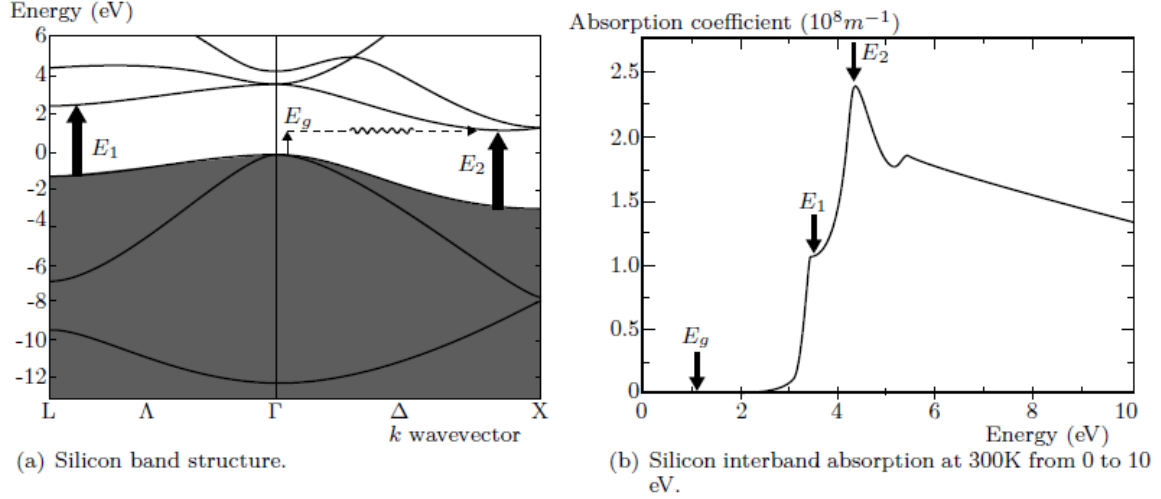


Figure 2.4: Absorption coefficient of silicon. (left) : band-to-band transition. (right) : absorption coefficient [13]

Analogously, **Light emission** also implies the two quasi-particles to respect the crystalline momentum conservation. In this process, an electron in conduction band returns in its fundamental state (located in valence band) and recombines with a hole in valence band by emitting a photon (for semiconductors with a bandgap energy larger than 0.5 eV). Wavelength of emitted photons are determined by the bandgap E_g and the energy of implied phonon (five different photon emissions are shown by the coloured arrows). If a phonon is absorbed during photon emission, the photon energy exceed the bandgap energy (blue arrow), and if emitted, it is the reverse (green, purple, orange and red arrows). Light emission is the basic concept for light-emitting photodiodes *LED*.

2.1.3 Mechanical properties of single crystal silicon

Now that the background about the *optoelectronic* part is given, let us shift on the *strain* one, related to mechanical properties of single crystal silicon. Those properties are based on the atomic bonding and crystal symmetry [8]. Silicon being an anisotropic material, its deformation and fracture properties depends on the orientation of the wafer.

²The absorption at those energy peaks are justified by the particular behaviour of the joined density of states *JDOS* in the case of parallelism existing between valence and conduction band. the *JDOS* is a quantum concept providing a measurement about the number of allowed transitions between occupied valence band and unoccupied conduction band

Young's modulus and Poisson ratio

Young's modulus and Poisson ratio are properties relative to interatomic elastic forces between atomic constituent of a solid. Hopcroft et al. [14] mentioned that **Young's modulus** of single-crystal silicon can differ up to 45 % by changing the orientation of the wafer (going from 130 to 188 GPa for bulk silicon). Analogous observation is made for **Poisson ratio** sweeping a range from 0.04 to 0.48. The derivation of those values is performed by using the elasticity formalism based on Hooke's law : $\sigma_{ij} = c_{ijkl}\epsilon_{kl}$, where c the 4-rank compliance tensor permits to link the σ to ϵ , the 2-rank stress and strain tensors. Silicon being a cubic system and the shear condition being equivalent, compliance tensor owns only 3 independent components : c_{11} , c_{12} and c_{44} . For any direction, Young's modulus and Poisson ratio can be derived based on the compliant tensor coefficients (Figure 2.5). Elastic constants also slightly varies with doping : its has been proved that a strong dopping decreases the Young's modulus value from 1 to 3 %.

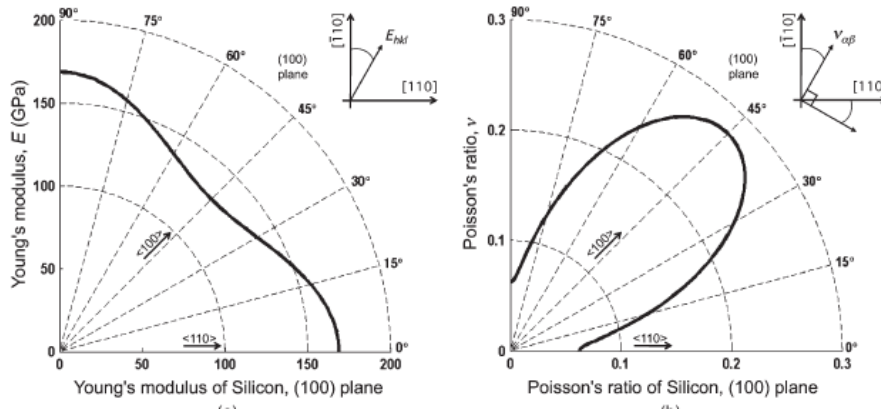


Figure 2.5: Young's modulus and Poisson ratio of silicon as a function of the crystallographic orientation [14]

Some investigations performed on silicon nanobeams point out a dependence of Young's modulus with dimensions at nanoscale. Bhaskar and al. mentions a decrease of 5% compared to bulk value (169 GPa is basically taken as average) of for a 200 nm-thick silicon film and a decrease of 40% for a 50nm-thick film [8]. Such huge changes cannot be explained simply by the increase of surface reconstruction phenomena. Other justification, based on the increasing influence of native oxide or volumetric defects, may explain this reduction which is until now not fully understood.

Fracture strength and strain Fracture strength of silicon is very dependent on the loading mode and defect population. This explains the wide range of values for this parameter found in literature (from 300 MPa to 17 GPa). For silicon nanoobjects, maximum fracture strain for uniaxial loading is located around 10% (obtained for silicon nanowires [15]). It is known from macroscopic metal sheet tensile testing that biaxial fracture strain is lower than uniaxial one, so the maximum biaxial fracture strain should not exceed this value of 5%. Literature suffers from a lack of information about silicon biaxial fracture strength.

Preferential directions for fracture propagation Silicon fracture occurs preferentially along $\langle 110 \rangle$ direction, by the cleavage on $\{111\}$ planes, and can also occur along $\langle 100 \rangle$ or $\langle 111 \rangle$ directions by the cleavage on $\{110\}$ planes}. Indeed, those plane families own the lowest surface energies (estimated at 1.19 J/m^2 for $\{111\}$ planes and at 1.50 J/m^2 for $\{110\}$ planes) [16]. Getting fractures in $\{111\}$ plane along $\langle 110 \rangle$ direction is not surprising : silicon as a diamond-type arrangement (two entangled FCC lattices), and this particular plane is the most close-packed plane, presenting a large spacing between each other. Moreover, $\langle 110 \rangle$ is the most compact direction in the crystal.

2.2 Effect of strain on opto-electronic properties

The principle of strain engineering is to improve materials properties related to reciprocal lattice features, like charge carrier mobility or carriers band-to-band transition, by deforming the direct lattice.

Strain tensor expression

One can associate a second strain tensor to this deformation by using normal stress components (ϵ_{xx} , ϵ_{yy} and ϵ_{zz}) and shear stress components (ϵ_{xy} , ϵ_{yz} and ϵ_{xz}). A useful alternative way to express the strain tensor is to use its spheric/deviatoric decomposition :

$$\underline{\underline{\epsilon}} = \begin{bmatrix} \epsilon_{xx} & \epsilon_{xy} & \epsilon_{xz} \\ \epsilon_{yx} & \epsilon_{yy} & \epsilon_{yz} \\ \epsilon_{zx} & \epsilon_{zy} & \epsilon_{zz} \end{bmatrix} = \underbrace{\begin{bmatrix} \epsilon_M & 0 & 0 \\ 0 & \epsilon_M & 0 \\ 0 & 0 & \epsilon_M \end{bmatrix}}_{\text{Volumetric strain tensor}} + \underbrace{\begin{bmatrix} \frac{2\epsilon_{xx} - (\epsilon_{yy} + \epsilon_{zz})}{3} & 0 & 0 \\ 0 & \frac{2\epsilon_{yy} - (\epsilon_{xx} + \epsilon_{zz})}{3} & 0 \\ 0 & 0 & \frac{2\epsilon_{zz} - (\epsilon_{xx} + \epsilon_{yy})}{3} \end{bmatrix}}_{\text{Deviatoric strain tensor}} + \begin{bmatrix} 0 & \epsilon_{xy} & \epsilon_{xz} \\ \epsilon_{yx} & 0 & \epsilon_{yz} \\ \epsilon_{zx} & \epsilon_{zy} & 0 \end{bmatrix} \quad (2.5)$$

with ϵ_M the hydrostatic strain defined as :

$$\epsilon_M = \frac{\epsilon_{xx} + \epsilon_{yy} + \epsilon_{zz}}{3} \quad (2.6)$$

Let us consider the strain applied on a cubic crystal unit cell. This decomposition permits to evaluate to volume change of the unit cell through the first tensor (**volumetric strain tensor**) and the effect of shear strain through the second and third one, both traceless (their sum constitutes the **deviatoric strain tensor**) [17]. The diagonal one relates the change of lengths along the three axes and the zero diagonal one represents the rotation of the axes of an infinitesimal cube. In the case of a cubic crystal like silicon, the application of uniaxial stress along one of the three $\langle 100 \rangle$ axes induces a non zero first term of the deviatoric strain tensor. If stresses are applied along $\langle 110 \rangle$ or $\langle 111 \rangle$ axes, the second term of this deviatoric strain tensor takes a non zero value. This master thesis being dedicated to the investigation of biaxial stress of silicon in the (100) plane³ along the $[110]$ directions, both hydrostatic and shear strain components has to be taken into account.

³Top surface of the silicon wafer available in WINFAB being oriented in the (100) plane of silicon crystal

Effect of strain on optoelectronic properties of silicon

Reciprocal lattice being the Fourier transform of the direct lattice, a change in the direct crystal lattice modifies the reciprocal lattice and associated physical properties. Whereas the application of a volumetric strain has no influence on the crystal symmetry, the presence of a shear strain reduces the number of symmetry operation in the crystal cell. In the case of biaxial stress applied in the (100) plane, the crystal system shift from cubic to tetragonal, leading to a reduction of the symmetry operation number from 48 to 16 (8 for each sublattice).

The band structure being dependent on the crystal symmetry, reducing the symmetry in the lattice modify the bands and/or the gap value. Bouhassoune et al. showed that [18] :

- a volumetric deformation induces a change of bandgap energy and shifts vertically the band edges. Indeed, the three axis of the cell being modify in an equivalent way, the system obtained after deformation remains cubic so symmetry is not reduces (degeneracies are not lift). Only lattice parameter a_0 changes.
- the presence of shear strain leaves the degeneracies existing in valence and conduction bands due to the symmetry breaking. A volumetric compressive deformation reduces the bandgap, and a tensile one increases it.

Degeneracy lift in silicon valence band though shear strain

As seen in the silicon band structure analysis, valence band at the Γ has a degeneracy order of 3. The application of uniaxial or biaxial strain (inducing a shear strain by definition) lifts this degeneracy leading to separates heavy-hole band and light hole-band. This is represented in **Figure 2.6**.

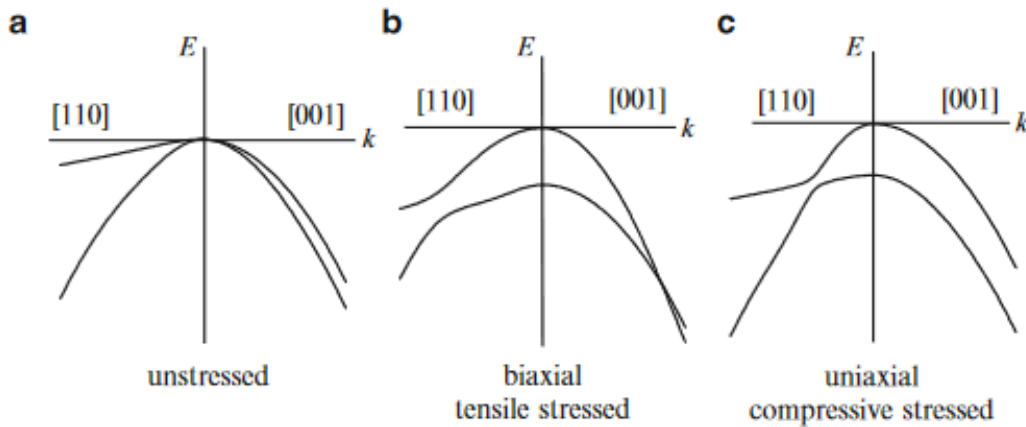


Figure 2.6: Degeneracy lift at the Γ point in valence band between heavy and light hole band. (a) : unstressed case. (b) : Biaxial tensile stressed case. (c) uniaxial compressive stressed case [17].

Degeneracy lift in silicon conduction band through shear strain

The Δ_6 star degeneracy in silicon conduction band is lifted if the deviatoric stress tensor is non zero. In such case, the cell undergoes some shear strain and the symmetry operations existing between the k-points related to each degenerate valley Δ are broken : the valleys are no equivalent any more and the 6-fold degeneracy is left. In the case of a biaxial strain applied in the (100) plane, the Δ_6 valleys are split into four higher Δ_4 valley along [100] and [010] directions, and two lower Δ_2 valleys along [001] (see **Figure 2.7, left**). This valley splitting is visible on band structure diagram of 1% biaxial strained silicon in (100) plane (**Figure 2.7, right**). One notices that the applied strain here decreases the bandgap.

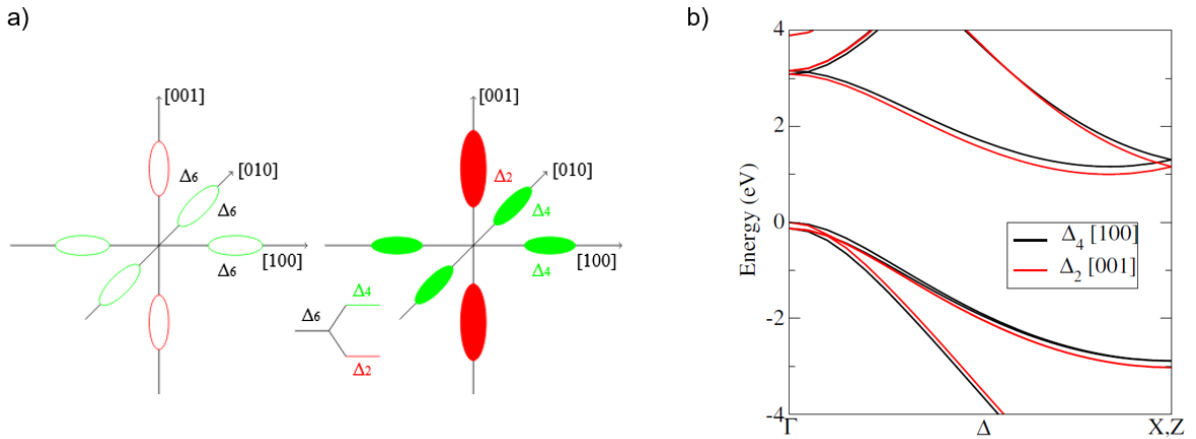


Figure 2.7: Degeneracy lift of the 6 equivalent Δ_6 valleys in conduction band through biaxial strain application in the xy plane. a) Δ_6 valleys splitting into Δ_4 and Δ_2 . b) Ab initio calculation of band structure of 1% strained silicon (in (100) plane [18]).

Take home message about strain engineering

To sum up, volumetric strain permits to change the bandgap energy by keeping symmetry existing in cubic system. A non zero deviatoric strain tensor (occurring in the presence of shear strain components ϵ_{xy} , ϵ_{yz} or ϵ_{xz} or non equal normal components ϵ_{xx} , ϵ_{yy} and ϵ_{zz}) induces some shear strain in the structure, the crystal symmetry is reduced (for instance from cubic to tetragonal system if a biaxial stress is applied in the (100) plane if silicon is considered). It follows band degeneracy lifts in valence and conduction **Figure 2.8. This justifies why monocrystalline silicon is used here** : a lattice with symmetries is needed to induce these changes, amorphous silicon is so not suited.

In addition to induce lifting degeneracies, shear strain also permits to shift the position of conduction band minimum in the Brillouin zone [19]. Such tuning modify the band-to-band transition process and the energy of the involved phonons. A indirect tot direct bandgap transition has been achieved for Germanium, enhancing tremendously its efficiency in optoelectronics devices like LED⁴. Strain can also modify the band curvature,

⁴Direct bandgap semiconductors are often preferred than indirect one, because the recombinaison

so the effective mass of charge carrier. Strain engineering dedicated to CMOS technology struggles to modify this band curvature to decrease as much as possible the charge carrier effective mass, increasing the charge carrier mobility.

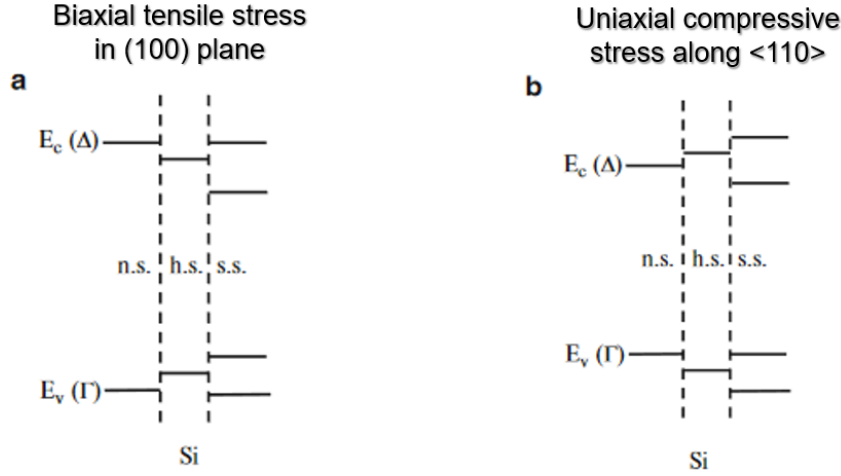


Figure 2.8: Diagrams of band splitting of Si under (a) biaxial tensile stress in the (100) plane. (b) $\langle 110 \rangle$ uniaxial compressive stress. *n.s.* refers to unstrained band, *h.s.* refers to hydrostatic strain and *s.s.* label the effect of shear strain [17]

2.3 Existing biaxial loading structures

To emphasize the advantages brought through on-chip materials properties characterisation at nanoscale, one has to review the existing techniques dedicated to thin film biaxial loading. It appears that far less methods are developed for biaxial loading of nano-objects than for uniaxial⁵. This can be explained mainly by the high complexity of accurate alignment between specimen and stress application direction. Nano-objects loading devices can be splitted in two categories : the external actuation devices and the internal actuation devices.

2.3.1 External actuation devices

The loading requires an external actuation, most of time being based on piezoelectric effect or electrostatic interaction, or other load cells. Here is a non exhaustive list of such techniques suitable for biaxial loading by external actuation :

- **Bulge test** : Bulge test is a widely used method consisting in applying a biaxial load on thin-films and evaluating the biaxial elastic moduli of materials by a deflection measurement [21]. In this technique, freestanding thin films are obtained by

process does not include electron-phonon interaction to fulfill conservation of momentum. The need of an electron-phonon interaction for the radiative recombination makes it less probable.

⁵Clearly emphasised in the review performed by Ozaki in 2018 [20]

performing a back etching of a selected region (with a rectangular or circular shape) in the substrate. Then the deflection of the suspended part is obtained by applying a uniform gas pressure to the film with a gas or a fluid. A maximum deflection (and strain) is obtained in the center of the membrane (**Figure 2.9**). The deflection of the membrane is then measured by interferometry. Material properties can be extracted by injecting this deflection into finite element analysis models. Bulge test is sometimes used to measure fracture strength, however fracture is often observed near the membrane edge, where the stress field is almost uniaxial, so the obtained values do not represent the real biaxial fracture strength. The great advantage of this technique is the simplicity of fabrication process and the ease handling of created structure. However, bulge test presents some cons, like the complex models needed to precisely define boundary conditions, or a high sensitivity of the results to slight variations of the sample dimensions. [22]

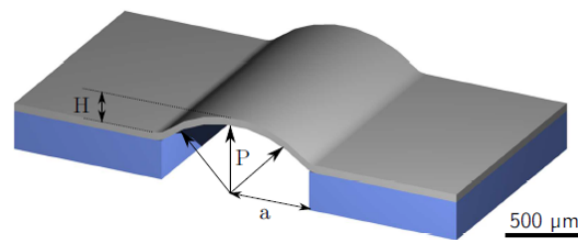


Figure 2.9: Deflection of a rectangular thin film during bulge test [22].

- **Nanoindentation** : nanoindentation usually permits to extract elastic and plastic properties of thin films by penetration of a tip with precise geometry (pyramidal, spherical, triangular...)[23]. The main drawback of this technique for elastic properties characterisation of very thin film is the difficulty to avoid substrate effect. In 2018, Ozaki et al. developed an on-chip device devoted to flexural strength characterisation of thin films. They pushed the center of free-standing circular membranes by a conical indenter until the membrane fracture on the reverse side of the specimen ⁶. The axisymmetry of the structure permits to induce an ideal biaxial tensile stress in the reversal side where fracture occurs. This method owns many advantages : no gripping or manipulation of micro-objects needed, inducing an high degree of reproductibility and simplicity. Furthermore, fracture has a high probability to occur at the center of the sample and not near the edges, like it is the case in bulge test (**Figure 2.10 (right)**) [20]. However, good alignment between indenter and sample is quite complex to obtain.
- **In plane biaxial tensile testing** : This method is based on the biaxial tensile testing of macroscopic metal sheets. A specimen with cruciform shape has its four extremities attached to external actuator, which simultaneously apply a force in perpendicular direction. Namazu et al. developed a biaxial tensile testing device for thin film specimen, using piezoelectric actuators (PZT). They used Raman spectroscopy to evaluate the level of strain reached. The specimen has a square central region of 300 μm length and a thickness of 5 μm . The maximum stress

⁶this deviates slightly from traditional indentation but it is worthy to mention this technique

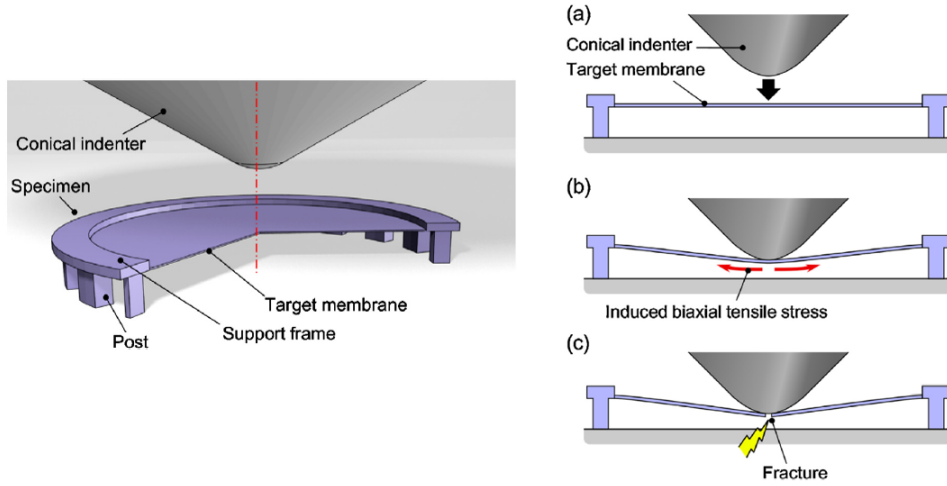


Figure 2.10: Flexural strength measurement by nanoindenter : (a) Alignment of conical indenter axis with center of the target membrane. (b) The indenter push into the membrane inducing biaxial stress. (c) When the stress exceeds the biaxial strength of the membrane, fracture occurs. [20].

reached during experimentation was around 150 MPa (they did not go until rupture) [24]. Such tensile testing structures based on macroscopic deformation encounters problems like sample manipulation, gripping and misalignment. Other drawbacks of these external loading methods are to involve a power supply and require a calibration of the loading cells.

2.3.2 Internal actuation devices

The advantage of internal loading techniques is the application of stress without any external equipment, suppressing the calibration procedure of equipments, specimen handling and other drawbacks. Two kinds of internal actuation exist, both based on the concept of *internal stress transmission* :

- **Hetero-actuated devices** : This actuation technique relies on the growth process of a particular material, called *actuator*, on the layer to deform. Thanks to the lattice constant mismatch or the difference of thermal coefficient of the two materials, the actuator owns "*internal stress*", which can be either tensile or compressive. LPCVD silicon nitride [8] and Nickel [25] have for instance around 1 GPa of internal tensile and compressive stress, respectively. By patterning this actuator and the target specimen, its possible to create regions were the thin-film specimen and the actuator are free-standing, giving the actuator the ability to transfer its internal stress to put specimen under strain. As biaxial loading is concerned, literature appears to be rather weak concerning internal actuation for material characterisation. Shchepetov et al [6] reports the fabrication and characterisation of an high quality ultra-thin silicon membranes (thicknesses of 6 nm, 27 nm and 54 nm) put under strain. The structure is built from a SOI wafer, on which a silicon nitride layer *SiN* is deposited by low pressure chemical vapour depostion (LPCVD). A window is opened in the nitride layer by UV-lithography followed by a dry etching, and finally a back etching

is performed to release the membranes. The structure is shown in Figure 2.12(left). Due to the internal stress induced by LPCVD process, the SiN frame around the free silicon window pulls on it by keeping the silicon film flat, avoiding any buckling. The strain level is controlled by tuning two dimensions of the structure : the width of the opened window w_m and the width between the edge of this window and the non free-standing part of the structure is w_c . The total width of the free standing part, w_s goes from 700 to 1700 μm . This investigations leads to strain from 0 to 0.15%, measured by Raman spectroscopy.

Fabregue and UCLouvain research team mentioned the fabrication of such biaxial loading structure for material characterisation (applied to aluminium thin-film in the paper [26]) based on internal stress transmission. A SEM image of such structures is shown in Figure 2.12(right).

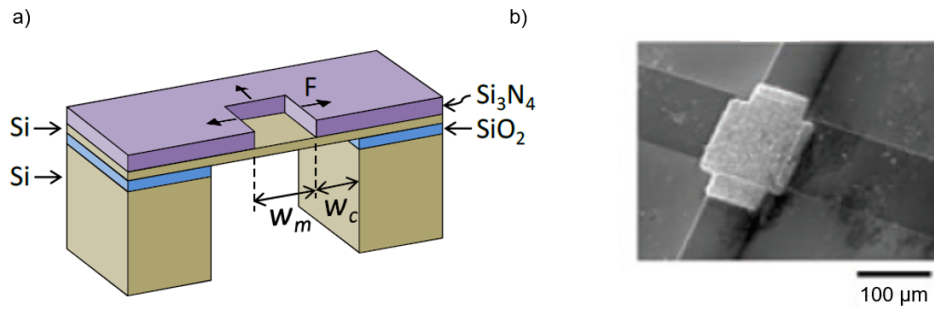


Figure 2.11: (left) Schematic representation of the free-standing structure proposed by Shchtepetov et al. Window is opened in the nitride layer with a width w_m , and the width between the edge of this window and the non free-standing part of the structure is w_c . F represents the internal stress of the SiN layer acting on the free silicon window [6]. (right) Biaxial tensile testing device developed by Fabregue et al.[26]

- **Auto-actuated devices** : the same material constitutes the specimen and the actuator. Actuator can thus be seen as a specimen prolongation attached to a fixed frame. Specimen and actuator layer being the same, the potential problem of overlap, adhesion and alignments between components are avoided. Auto-actuation is only possible if the thin-film owns internal stress. Gassenq et al. used this approach to apply biaxial strain on free-standing germanium thin-films [27]. It consists in patterning the top-Germanium layer of a $GeOI$ wafer to create cruciform shape, similar to the one presented in the last external actuation technique. The stress transmission occurs after etching the sacrificial layer. It permits to reach a 1.9% biaxial strain corresponding to the indirect to direct transition of bandgap for germanium.

2.4 Concept of lab on-chip

The advent of MEMS and NEMS permits tremendous enhancement in the field of micro- and nanofabrication techniques. Their present maturity and stability makes such techniques usable, and even appealing, for a broad range of applications, like materials

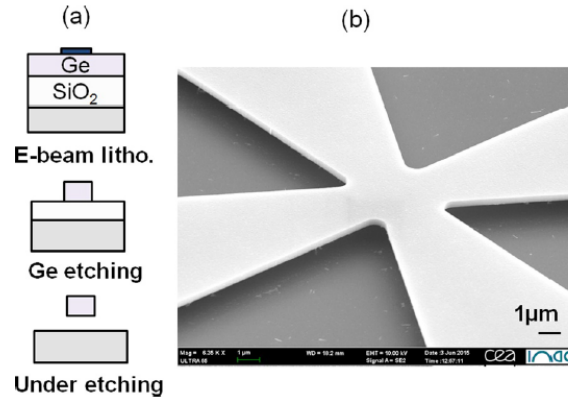


Figure 2.12: Free-standing Germanium membrane after patterning and release. On right a brief summary of the fabrication process [27].

characterisation at nanoscale. New testing devices were thus imagined by scientist community, based on micro and nanofabrication techniques, like the Shchepetov and Gassenq ones presented in the previous section.

For almost 15 years [26], UCLouvain decided to come on board and to develop a particular nanomechanical testing device, fabricated using top-down approach. These structures, called **labs on-chip**, permits the measurement of the mechanical response and the characterisation of other physical properties of nano-objects like thin-films, nanobeams, nanowires and nanoribbons for various materials : aluminium, palladium, silicon... Labs on-chip were developed for various thermo-mechanical properties investigations, including elasticity response, strain-hardening, creep, fracture mechanism investigation with the *cracks on-chip*...) and different loading modes (uniaxial and shear).

The lab on-chip is based on the same principle than the internal actuation devices introduced previously : the use of the internal stress created during a film deposition on a layer to deform it. Here is a non exhaustive list demonstrating how powerful is this characterisation method :

- Labs on-chip benefits from the micro and nanofabrication techniques, providing a **high reproducibility** between structures.
- Labs on-chip being generally fabricated on 3 inches wafers (sometimes even larger), thousands of structures can be fabricated simultaneously, meaning **high throughput capabilities**.
- A **large range of strain** can be obtained by controlling the progressive variation of labs dimensions. Each set of dimensions corresponds to one point on the stress/strain curve. Each specific structure being reproduced a certain number of times on the wafer, this makes possible **statistical analysis**.
- Possible issues linked to external actuation devices like the monitoring the loading (gripping, calibration...) are avoided.

- The lab on chip consists in a multi-purpose testing device, to permit the extraction of several materials properties with the same structures : mechanical (like stress/strain curves, fracture strength, creep, strain-hardening...), electrical (like piezoresistivity, charge carrier density) etc. by various characterisation method : optical characterisation through SEM observation, Raman spectroscopy, electrical probing, Hall measurement...
- Some labs on-chip are **compatible with in-situ TEM observation and TEM straining**, for a better understanding of deformation and fracture process occurring at nanoscopic scale (they require a process differing slightly from the classical one, implying a window opening in the back face of the wafer called *back etching*).
- By giving a certain shape (trapezoidal) to the actuators, the loading rate can be controlled

However, this method owns a few drawbacks :

- Some analysis, like SEM measurements, are performed after fabrication, when a **possible relaxation** could have already take place (not the case for silicon)
- Fabrication process of a lab on-chip is relatively long and includes many critical steps, like photolithography mask alignments, etching or release. The back-etching for transmission measurements adds even more complexity in the process.

The biaxial tensile testing stage developed in this master thesis is based on the same principle than those produced previously at UCLouvain. A general overview of the process dedicated to lab on-chip creation is shown in **Figure 2.13**. Firstly, a sacrificial layer is deposited on a substrate, usually a standard silicon wafer. The specimen layer is then deposited on this sacrificial layer and is patterned. The actuator layer is deposited on the specimen one⁷ and patterned, by ensuring to have some overlap region between specimen and actuator. The sacrificial layer is then removed during a step called *release*, to make specimens and actuators free-standing, allowing the internal stress transfer. The mechanical strain in the specimen is then obtained by SEM measurement of the displacement u between some fixed and moving cursors.

The material of each layer has to be chosen carefully based on the layer specifications. The sacrificial layer for instance is convenient if it exists an etchant removing this sacrificial layer without degrading too much the actuator and the specimen layers. The actuator has to own a high level of tensile internal stress, with the lowest internal stress gradient possible over its cross section to avoid out-of-plane deformation. It is also important to choose actuator material with a fracture strength larger than the specimen one, to ensure that the specimen breaks first, to get information about specimen fracture strength and also to extract all the stress/strain curve of the sample. The actuator must not be prone to creep and has to own a linear elastic behaviour to permit analytical development for structure dimensioning. The specimen material is imposed by the investigation.

⁷The specimen and actuator deposition steps can be inverted

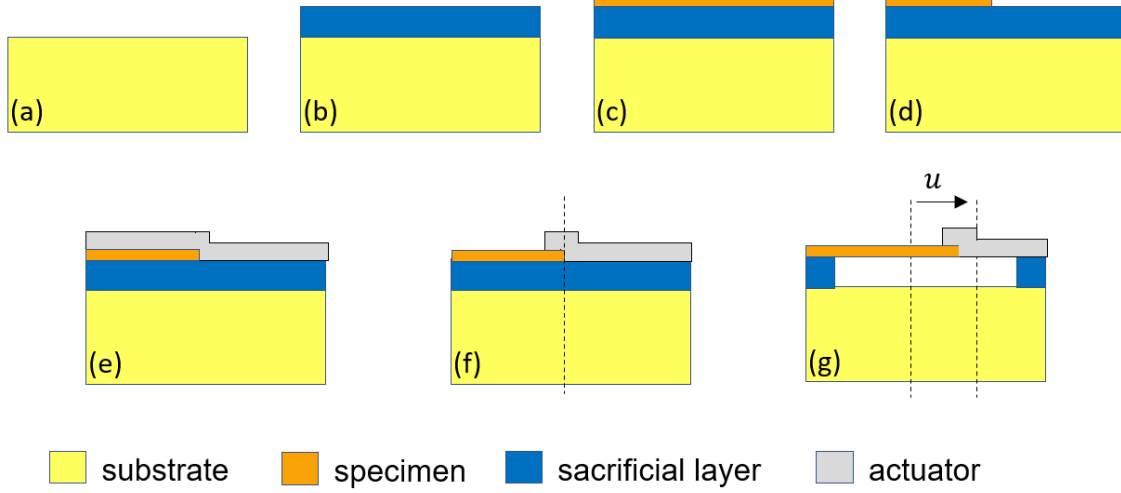


Figure 2.13: Schematic representation of usual fabrication process for lab-on chip. (a) initial substrate. (b) sacrificial layer deposition. (c) Specimen layer deposition. (d) specimen layer patterning. (e) : actuator layer deposition. (f) : actuator layer patterning. (g) : sacrificial layer etching. Inspired of [22]

Labs on-chip devoted to uniaxial tensile testing of silicon nanowires are shown as example in Figure 2.14. These structures permitted to extract the Young modulus, fracture strength and piezoresistance. The last picture shows an set of different structures with various actuator lengths.

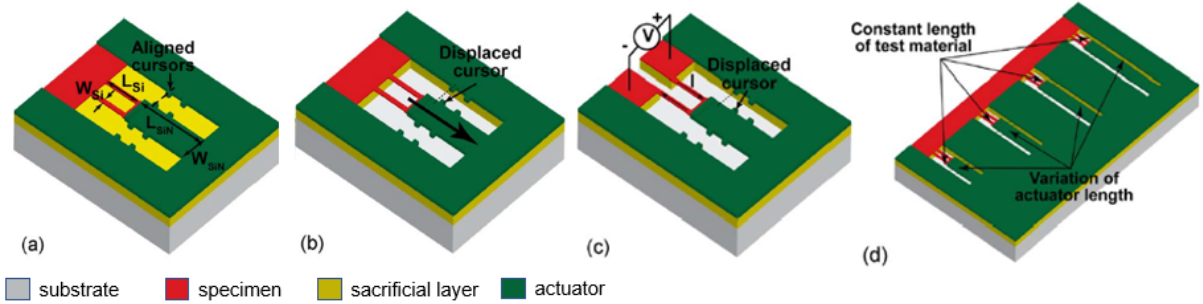


Figure 2.14: Lab on-chip developed by Bashkar et al. to put Si nanowires of width W_{Si} and length L_{Si} under strain by using silicon nitride beams (width W_{SiN} and length L_{SiN}). (a) Structure before release. (b) Structure after release. (c) Electrical measurements performed after release. (d) Tensile testing structures with different actuator length. [8]

2.5 Characterization tool : Scanning electron microscope

Strain in lab on-chip characterisation is generally obtained via two ways : the Raman spectroscopy and the scanning electron microscopy. The first way being not used in this

master thesis, only the SEM working principle is introduced here.

Displacement of the smallest structures is expected to be about tens of nanometers. Optical microscopy does not provide resolution large enough to measure them, so one has to shift to electron microscopy. SEM, or scanning electron microscope, is a non-destructive technique consisting in scanning the surface with a high energy electron beam to image the surface topography or composition. SEM resolution is nowadays between 1 and 20 nm [28]. Its working principle is based on the electron-matter interaction : when an electron beam hits a surface, an energy redistribution occurs in the surface matter. Due to some inelastic interactions between the electron beam and the sample surface, some low energy electrons (typically between 0 and 50 eV [29]), called secondary electrons, are emitted from the surface. These electrons are detected by a secondary electrons detector, which creates and amplified signal for each point hit by the electron beam. The nature and the topography of the surface determines the signal intensity. A basic SEM is composed of an electron gun to create the electron beam, a set of electron lens (coils) coils to focus it, a sample stage, one or several detectors and a signal treatment system (Figure 2.15). Gas atmosphere attenuating the electron beams, vacuum is needed for classic SEM⁸ (below 10^{-4} Torr to efficiently work).

A disadvantage of the SEM comes from the charging effects when one works with insulating materials, like silicon nitride or silicon oxide. Indeed, without charge evacuation, electrons accumulates at the surface and induces some distortions of the electron beam. A field emission gun has thus to be used to perform accurate imaging of these materials.

⁸Some particular SEMs, called environmental SEM or ESEM, are dedicated to imaging of wet or uncoated specimens and has thus the particularity to work under gas and no in vacuum conditions [30]

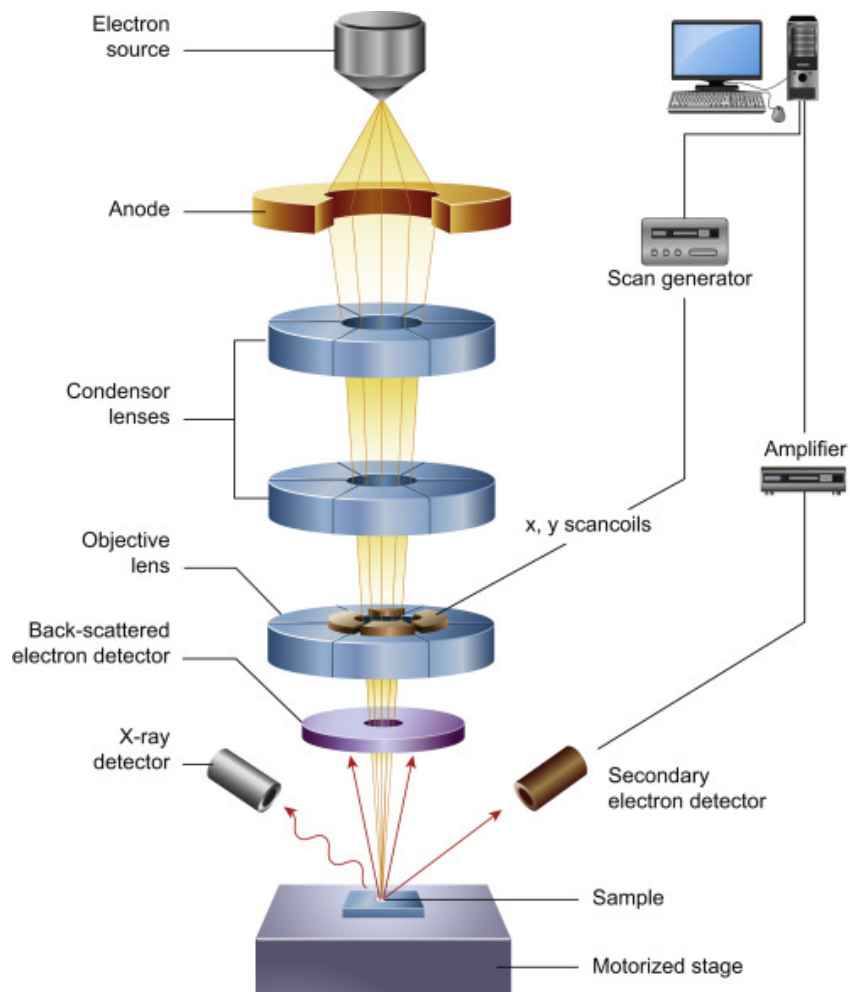


Figure 2.15: Main components of a classic SEM [31]

Chapter 3

Design of biaxially deformed silicon structures

This chapter is devoted to design a lab on-chip convenient for silicon thin-film biaxial tensile testing. The main difference between this structure and the ones devoted to uniaxial tensile structures is the patterning of the thin-film, the working principle remains exactly the same. The design being dependent on the materials constants of the different layers, one has to perform a material choice concerning actuator and sacrificial layer to use the right parameters values. A simple method to estimate the strain reached with a certain set of dimensions is based on analytical development. Even though it requires some hypothesis, sometimes quite strong, it gives an insight about the parameters allowing to sweep a wide range of strains. The first part of the design is dedicated to the development of an analytical model based on linear elasticity theory. In opposition to uniaxial tensile testing specimen when one deals with simple rectangular beams, the specimen dedicated to biaxial tensile testing are far much complex. Macroscopic biaxial tensile testing specimens devoted to metal thin sheet characterisation usually have a cruciform design, like the one used by Gassenq in the state of the art. The main inconvenient of those structures is the non uniform stress and stress distribution in the specimen, due to presence of high stress region. A set of cruciform design is thus investigated by finite elements analysis (FEA) in the aim to deduce the ones minimizing at best these effects. Once selected, finite element simulation are used in a second purpose : confirming the trends observed in analytical development and perform a selection of dimensions to sweep a range of strains, going from 0 to 4%. Indeed, it is well known that fracture strain under biaxial loading is lower than under uniaxial solicitation. Fracture strains of nanobeams reaching at maximum around 5%, investigating strains up to 4% seems convenient.

Materials choice for design

As it has been seen previously, each layer has some specifications concerning the material. The material selection for layers is already quite optimised due to the experience of UCLouvain workers in the on-chip domain, so these materials were selected for design. Here are the summarized justifications for these choices. A good material for **sacrificial layer** is a material presenting a good selectivity¹ compared to silicon for a particular

¹selectivity is defined as the ratio of etching rate between two materials in a particular etchant.

etchant. Silicon is known to present an extremely good resistance in fluoridric acid (HF), whereas silicon oxide SiO_2 not. The selectivity between Si and SiO_2 in HF is around 1:1000. Actuator material has also to exhibit a good selectivity to HF , this criteria has also to be kept in mind during its choice.

A suited and straightforward practice in the lab on-chip design devoted to silicon characterisation is to use SOI (silicon-on-insulator) technology to be exempt of the sacrificial layer growth and thin film deposition. SOI consists in a stacking of three layers : a silicon substrate, a buried silicon oxide layer (BOX) and a top-silicon layer. This also ensures to work with a high quality monocrystalline silicon thin film.

As actuator material is concerned, a usual candidate is the LPCVD silicon nitride. This material can reach tensile internal stress around 1.1 GPa and present not stress internal gradient over its cross-section, exhibits a linear elastic behaviour, is inert thus induces nearly no restriction concerning the process steps in *WINFAB* facilities. It also presents an acceptable resistance to HF .

The set of material being selected, their average values for mechanical constants can be selected in literature. The analytical model can now be derived.

3.1 Derivation of a simplified linear elasticity theory-based model

The aim of this section is to derive a general equation predicting the displacement of the released structure as a function of its dimensions, based on the elasticity theory. Once done, one can have an insight about which geometrical parameter variations have the ability to significantly modify the strain level reached in the film. This is crucial to sweep efficiently the range of desired strains.

3.1.1 Definition of the simplified system

The stress and strain induced in the specimen can be theoretically deduced by using solid linear elasticity theory. Equations related to biaxial loading of the simplest cruciform shape being complex to derive, it has to be simplified. Gassenq, Namazu and Fabregue [27],[24],[26] opted to link the four extremities of the cross-shaped specimen to an actuator (like in [Figure 2.12 \(right\)](#)). An alternative approach consists in linking one specimen extremity to one actuator in each direction at one extremity, while the other is fixed to an unmovable frame. In this way, only one actuator is responsible of the total displacement in each direction. A simplification for analytical treatment is to restrict the investigation to the central part of the cross, a rectangular thin-film of initial length $L_{Si,0}^x$ and width $L_{Si,0}^y$. The system is depicted in [Figure 3.1 \(left\)](#).

The model developed in this section is based on the equilibrium conditions between components after release. The release here consists in the wet etching of the sacrificial

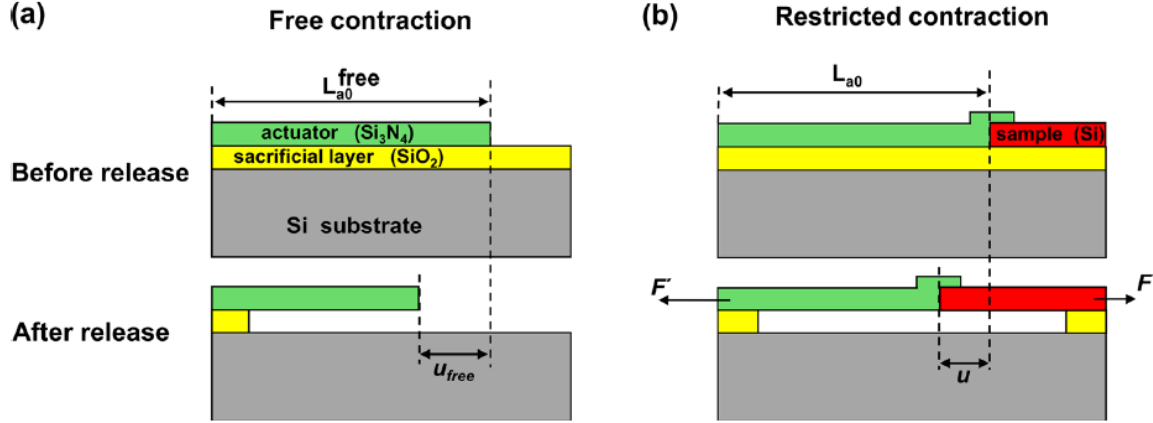


Figure 3.2: Schematic representation of the general model. Upper pictures represents the situation of a free actuator (left) and an actuator attached to a silicon sample (right) before release. Down pictures represents these two system after released, being now free-standing. The free actuator relaxes its internal stress by undergoing a displacement of u_{free} , whereas in the actuator-silicon structure, the actuator free release is thwarted by the presence of silicon, reaching after release a displacement u . [7]

3.1.2 Hypothesis

At first sight, one might be sceptical about the use of a continuum mechanics-based theory for a nanoscopic system. Indeed, the thickness of the components here is limited to a few hundreds of nanometers. By reminding that one atomic plane has a thickness around 0.3 nm, for a 300 nm layer, one works with 1000 atomic planes. One could so consider that the atomic fluctuations are almost smeared out by the number of atoms, leading to a convenient modelling.

Three main hypothesis are set to perform present investigation :

- One assumes that deformations remain in the elastic domain, which permits to link stresses and strains by a linear relation.
- The components are considered to be homogeneous and to present an isotropic behaviour (whereas it is true for amorphous components like silicon nitride, it has been seen in the state of the art that Young's modulus (Poisson ratio too) of silicon can change up to 45% with the crystallographic orientation).
- Purely biaxial stress and to neglect edge and corners effects, leading to a uniform strain distribution in the specimen (it is not really the case due the presence of two successive anchored extremities of the film inducing a tensile region in one corner, but let us consider this hypothesis valid in first approximation).

3.1.3 Model derivation

The starting point is to express properly the stresses σ_a^x and σ_a^y applied by actuators on the specimen in each direction. Assuming that actuators have a Young's modulus E_a , the stress of the actuator transmitted to the specimen can be expressed by Hooke's law. The strain considered here is at intermediate state between 0 (unreleased actuator) and mismatch strain (free released actuator). It can be simply expressed by subtracting the mismatch strain ϵ_a^{mis} and the strain due to the internal stress transmission in the specimen, with displacement u and initial actuator length L_{a0} **Figure 3.2** :

$$\sigma_a^x = E_a \left[\epsilon_a^{mis} - \frac{u^x}{L_{a0}^x} \right] \text{ for the horizontal actuator} \quad (3.1)$$

$$\sigma_a^y = E_a \left[\epsilon_a^{mis} - \frac{u^y}{L_{a0}^y} \right] \text{ for the vertical actuator} \quad (3.2)$$

The general expression of the stress in the specimen can be derived in the same way (E_{Si} being silicon Young's modulus and ν_{Si} silicon Poisson ratio). The silicon sample being from blanket SOI, mismatch strain is not considered for this material. Using linear elasticity formalism, one obtains, by considering a rectangular film with initial dimensions $L_{Si,0}^x$ and $L_{Si,0}^y$ and the displacement reached at equilibrium in each direction u_x and u_y :

$$\sigma_{Si}^x = \frac{E_{Si}}{(1 + \nu_{Si})(1 - 2\nu_{Si})} \left((1 - \nu) \frac{u^x}{L_{Si,0}^x} + \nu \frac{u^y}{L_{Si,0}^y} \right) \quad (3.3)$$

$$\sigma_{Si}^y = \frac{E_{Si}}{(1 + \nu_{Si})(1 - 2\nu_{Si})} \left((1 - \nu) \frac{u^y}{L_{Si,0}^y} + \nu \frac{u^x}{L_{Si,0}^x} \right) \quad (3.4)$$

Equilibrium condition being based on force and not on stress, one has to convert those stresses in forces by dividing them by a cross section : for the silicon specimen those cross-sections are simply $S_{Si}^x = L_{Si,0}^y t_{Si}$ and $S_{Si}^y = L_{Si,0}^x t_{Si}$ (specimen cross section variation is considered as negligible here). The trapezoidal actuator shape is more complex to handle due to its variable cross section. To simplify the situation one can work with a rectangular actuator with the same length than the trapezoidal actuator and an effective width, depending on the length of large w_{max} and small w_0 basis. The derivation of this effective width is performed based on the computation of the general stiffness constant, where a trapezoidal actuator is considered as a succession of infinitesimal springs elements with variable cross sections put in series (development present in Appendix, [page 112](#)). This effective width w_{eff} can be expressed as :

$$w_{eff} = \frac{w_{max} - w_0}{\ln \left(\frac{w_{max}}{w_0} \right)} \quad (3.5)$$

The actuator cross section can be so expressed as $S_a^i = w_{eff}^j t_a$. The equilibrium being reached when the sum of forces in each direction cancels, one gets by equalizing F_{Si}^x to F_a^x

and F_{Si}^y to F_a^y :

$$\begin{cases} E_a w_{eff,a0}^x t_a \left[\epsilon_a^{mis} - \frac{u^x}{L_{a0}^x} \right] = \frac{E_{Si} L_{Si,0}^y t_{Si}}{(1+\nu_{Si})(1-2\nu_{Si})} \left((1-\nu) \frac{u^x}{L_{Si,0}^x} + \nu \frac{u^y}{L_{Si,0}^y} \right) \\ E_a w_{eff,a0}^y t_a \left[\epsilon_a^{mis} - \frac{u^y}{L_{a0}^y} \right] = \frac{E_{Si} L_{Si,0}^x t_{Si}}{(1+\nu_{Si})(1-2\nu_{Si})} \left((1-\nu) \frac{u^y}{L_{Si,0}^y} + \nu \frac{u^x}{L_{Si,0}^x} \right) \end{cases} \quad (3.6)$$

This linear system can be re-expressed by using matrix formalism :

$$\begin{pmatrix} \frac{E_a w_{eff,a0}^x t_a}{L_{a0}^x} + \frac{E_{Si}(1-\nu_{Si})L_{Si,0}^y t_{Si}}{(1+\nu_{Si})(1-2\nu_{Si})L_{Si,0}^x} & \frac{E_{Si}\nu_{Si}L_{Si,0}^y t_{Si}}{(1+\nu_{Si})(1-2\nu_{Si})L_{Si,0}^y} \\ \frac{E_{Si}\nu_{Si}L_{Si,0}^x t_{Si}}{(1+\nu_{Si})(1-2\nu_{Si})L_{Si,0}^x} & \frac{E_a w_{eff,a0}^y t_a}{L_{a0}^y} + \frac{E_{Si}(1-\nu_{Si})L_{Si,0}^x t_{Si}}{(1+\nu_{Si})(1-2\nu_{Si})L_{Si,0}^y} \end{pmatrix} \begin{pmatrix} u^x \\ u^y \end{pmatrix} = E_a \epsilon_a^{mis} \begin{pmatrix} w_{eff,a0}^x t_a \\ w_{eff,a0}^y t_a \end{pmatrix} \quad (3.7)$$

By solving numerically this system, the equilibrium displacements u_x and u_y can be obtained for any dimensions of specimen and actuator.

3.1.4 Dimension limits

Now that the model is derived, dimensions of components have to be discussed to present coherent results. Indeed, the integration, fabrication process or coupling between structure elements limit the choice of dimensions. Here is the range of dimensions estimated as acceptable for the lab on-chip :

- **Film length and width** : The release step fix the higher boundary concerning film length and width. The larger the specimen is, the larger has to be the actuator size, so the release time increases. Silicon nitride owning a non infinite selectivity in HF , this can be problematic³ Also, increasing the dimension increases the amount of matter, so the presence of defects. Fracture being initiated near defects, this reduces the fracture stain of the specimen. The lower boundary is linked to the characterisation by Raman spectroscopy : the laser spot definition being approximately a $1 \mu m^2$ area, taking too small length and width value will considerably reduce the number of point for local strain mapping. **A good compromise is to select films between $5 \mu m^2$ and $10 \mu m^2$.**
- **Film thickness** : This parameter depends mainly on the specifications of the investigation. Film thickness is restricted by the fabrication process, and can be variable to investigate properties modifications due to size effects. The lab on-chip being devoted to nanoscopic material characterisation, **sub-micrometer thick silicon specimens have to be considered.**
- **Actuator length and width** : the actuator dimensions are based on the film dimensions. Typically, the small basis overrides the cruciform specimen of $1 \mu m$ at each extremity, to avoid an eventual misalignment problem during the release. Actuators having a trapezoidal shape, their large basis has to be smaller than two times their length. Otherwise, consecutive actuators overlap. The actuator has to be designed to be fully released after the specimen. For large actuators, holes has to

³This element is almost devoted to narrow actuators. A solution for wider actuator can be to include some holes in the actuators to decrease this release time

be included to keep a convenient release time ⁴. The highest dimension for silicon nitride actuator being around 2000 μm in previous investigations, this value is set as higher boundary. Moreover, choosing higher values would decrease the integration.

- **Actuator thickness** : The actuator maximum thickness limited by the deposition technique. A rule of thumb for silicon nitride actuator deposited after patterning the specimen is to take an actuator thickness at least one half of the specimen thickness to ensure a good covering step with the specimen extremity. The quality of this covering also depends on the conformity of the deposition method. For LPCVD *SiN* deposition technique, the thickness limit is around 400 nm.

3.1.5 Graphical representation of results

The influence of geometrical parameters on strain can be represented either by **3D plot** or by **behaviour lines diagram**. Due to the huge number of geometrical variables, one has to fix some of them for graphical representation in both cases. Materials value for representation are $E_{Si} = 160$ GPa, $E_a = 240$ GPa [14], $\epsilon_a^{mis} = 0.00324$ [8]. In the following, the positive sign is chosen to represent tension and negative sign compression) and $\nu_{Si} = 0.22$.

3D plot representation

MATLAB software was used to solve the system (Equation (3.7)) and to represent the plots. An arbitrarily choice was to fix the film dimensions and to change the actuator ones. Film dimensions are set to $L_{Si,0}^x = L_{Si,0}^y = 10$ μm and $t_{Si} = 150$ nm. The small basis of actuator is thus chosen at 12 μm , to avoid misalignment problem during the release.

Figure 3.3 shows the level of strain and displacement obtained as a function of actuator length and width. Thickness is set at 400 nm. The actuator length is taken from 10 to 2000 μm , and the effective width from 15 to 320 μm , corresponding to a large basis from 15 to 1500 μm . It appears that sweeping a range of effective width by keeping length constant is more efficient than the reverse. By suggesting that biaxial fracture strain of monocrystalline silicon is lower than 5% [8], the strain levels associated to highest effective width (almost 0.2) is far above the strain range of interest here.

Figure 3.4 shows the level of strain and displacement obtained as a function of actuator length and thickness. The large basis is set at 50 μm and the small one at 15 μm ($w_{eff} = 27$ μm). The actuator length is taken from 10 to 2000 μm and the thickness from 10 to 400 nm. The range of strain obtained by keeping width constant is nearly 10 times smaller than by varying it. Figure 3.5 shows a cut in this graph for thicknesses 100, 200, 300 and 400 nm. Taking a large thickness permits firstly to reach higher strain levels, and secondly to sweep a broader strain range by varying length : by taking actuator length from the 10 to 500 μm , a [0.002;0.02] strain range can be obtained for a 400 nm-thick actuator, compared to a [0.002;0.006] for a 100 nm-thick one.

⁴position of these holes is investigated in FEA part

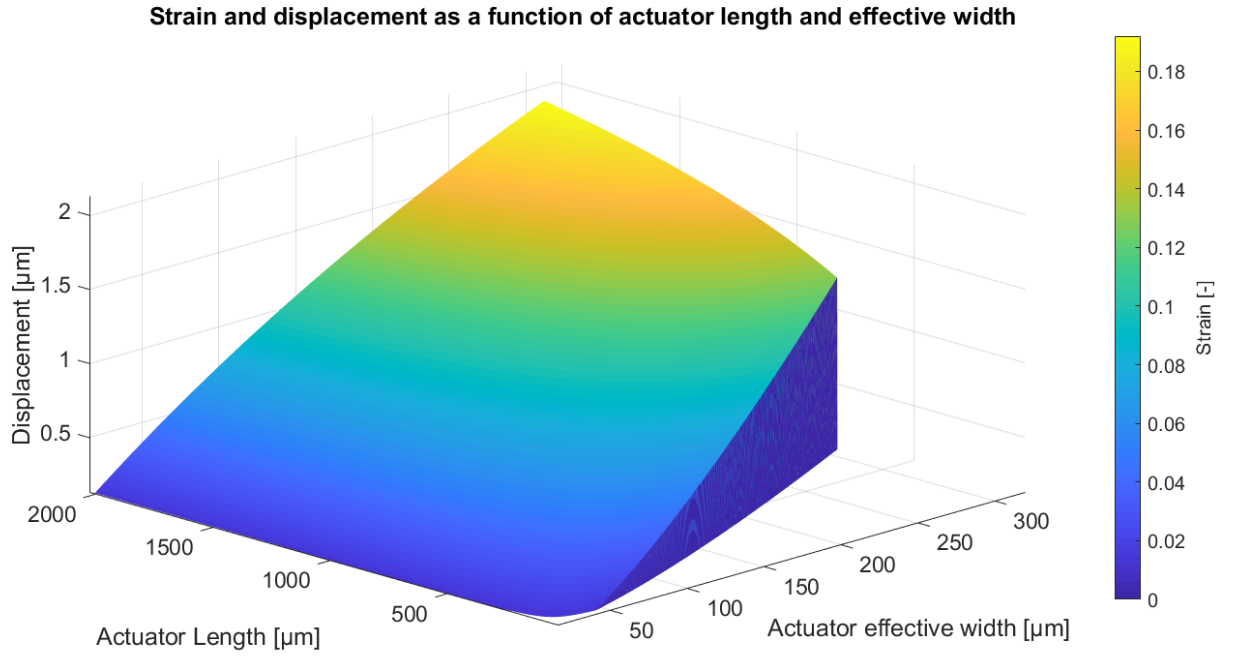


Figure 3.3: Displacement and strain reach depending on actuator length and effective width, actuator thickness being set to 400 nm.

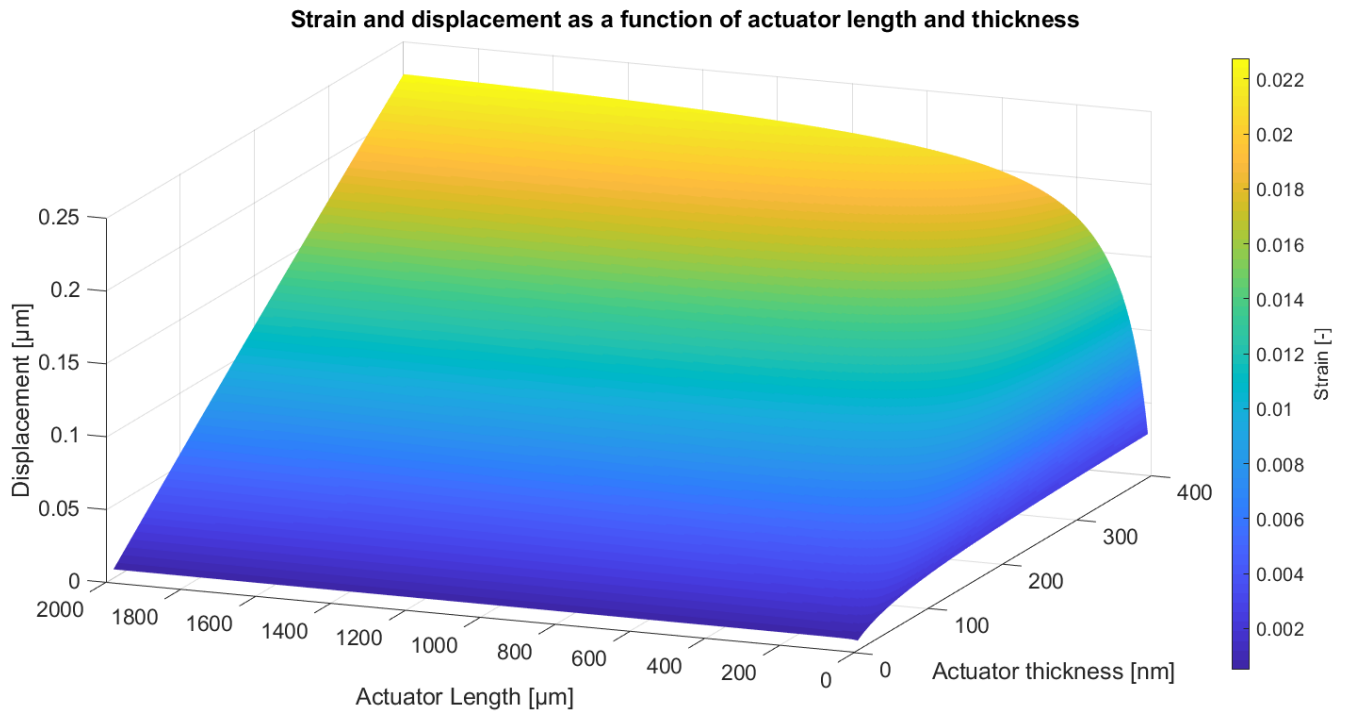


Figure 3.4: Displacement and strain reach depending on actuator length and thickness, effective width set to 27 μm (associated to a 12 μm small and 50 μm large basis).

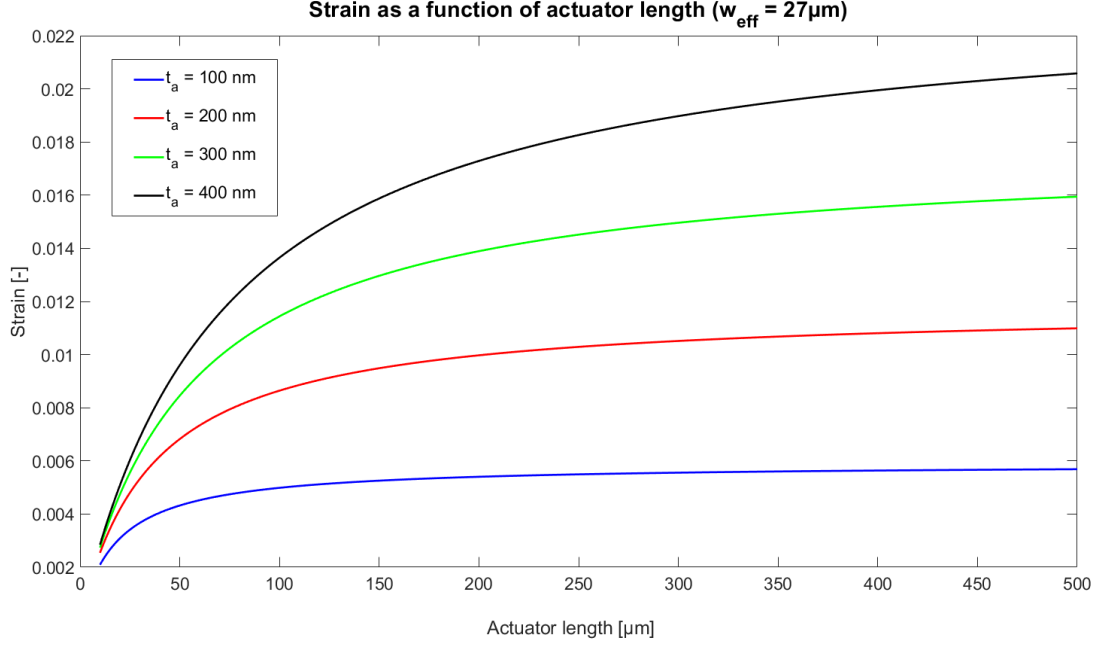


Figure 3.5: Strain as a function of actuator length for $w_{eff} = 27 \mu\text{m}$ and different actuator thicknesses

Behaviour lines representation

Behaviour lines representation is a usual method for lab on-chip dimensioning devoted to uniaxial or shear loading. It gives the same information than that obtained by solving the equation system (Equation (3.7)) but for discrete predefined sets of dimensions and does not require a numerical solver. This method consists in the construction of force/displacement lines for silicon thin film and silicon nitride actuator (see Figure 3.6). The film line represents the total force needed to apply to the silicon film to reach a precise displacement. This force is calculated for several points from a defined displacement range. It has a positive slope. The actuator line displays the total force given by the actuator as a function of its relaxation state, situated between 2 extreme case : being totally unreleased (no relaxation of its internal stress, so it undergoes zero displacement) and having undergone a free release (having reached a state without any internal stress, so undergoing a maximum displacement). It has a negative slope. Following first Newton law, the released system is at equilibrium when the total force given by the actuator is exactly the one needed to stretch the film. In this state, the interface between the components has undergone a displacement, called u , the equilibrium displacement. It can be obtained in the present method by the abscissa of the intersection between film and actuator lines. Lines depending on film and actuator dimensions, changing the dimensions of elements induces a change in the intersection position, so a change in the displacement reached at equilibrium too. This method provides so a direct way to predict the reached displacement of a particular set of actuator/film dimensions.

Thin film line construction The aim is to deduce the total force needed to apply in the two directions on a silicon film of fixed dimensions to obtained some desired displace-

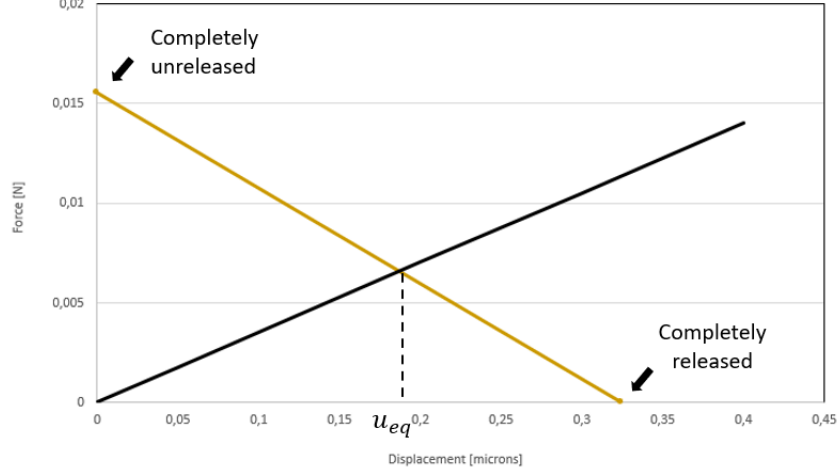


Figure 3.6: Behaviour lines overview. Positive slope line refers to a set of film dimensions. Negative slope line refers to a set of actuator dimensions

ments (associated to arbitrarily chosen strain levels). Only square films are considered for simplicity here.

For a set of film dimensions (length and thickness), 7 displacements are considered, each one associated to a particular level of strain in each direction : 0, 0.001, 0.005, 0.01, 0.02, 0.03 and 0.04. The associated stresses are deduced by Hooke's law, using same equations than presented before (Equation (3.3) and Equation (3.4)), and then are converted into force. Figure 3.7 shows the influence induced by film dimensions changes. As the film thickness decreases, the cross-section decreases, so the force increases (by reminding that stress only depends on material properties and defined strain level). The displacement (and associated force) increases linearly with the film length.

Actuator line construction The displacement associated to this free release corresponds to the X-intercept of the line in the graph. Strain mismatch and Young's modulus allow to deduce the stress of the fully unreleased structure : $\sigma_{max}^{act} = \epsilon_{max}^{act} E_{act} = 0.778$ GPa. The stress is multiplied by the cross section to get the total force in unreleased state, keeping the actuator effective width to compute the cross section.

The influence of actuator dimensions is shown in Figure 3.8. Actuator length influences the slope, so it comes as expected that longer the actuator is, larger is the displacement in the fully released state (X-intercept value). Actuator thickness and width have a quite similar influence on behaviour lines because both of them affect the cross section : their increase induce an increase of the force of completely unreleased actuator (Y-intercept value).

Lines intersection and equilibrium states By plotting several film and actuator lines, one is able to predict the equilibrium displacement for each possible set of film and actuator dimensions, following the above rules concerning the relevancy of each dimension.

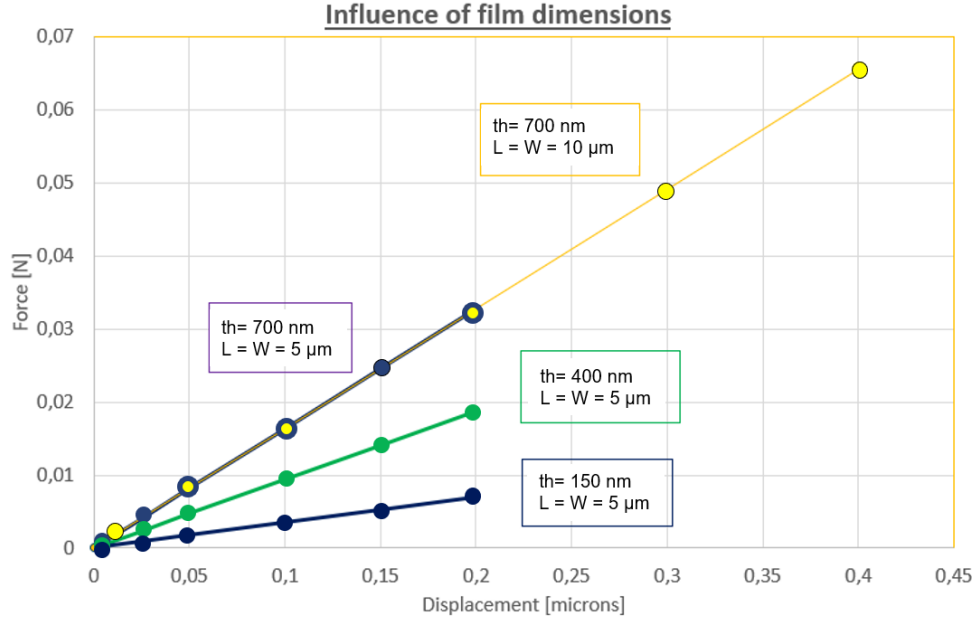


Figure 3.7: Thin film behaviour lines for a $5 \mu\text{m} \times 5 \mu\text{m}$ ($L \times W$) square film with a thickness th of 150, 400 and 700 nm and for a $10 \mu\text{m} \times 10 \mu\text{m}$ square film with a thickness of 700 nm. Each coloured dot corresponds to a precise strain level : (from left to right) : 0, 0.001, 0.005, 0.01, 0.02, 0.03 and 0.04. Yellow points surrounded by a blue circle belongs to both $5 \times 5 \mu\text{m}$ and $10 \times 10 \mu\text{m}$ film

In order to restrict the number of variable parameters, $5 \mu\text{m}$ and $10 \mu\text{m}$ edge square films are considered, with thicknesses of 150, 400 and 700 nm. As actuators are concerned, the selected thicknesses are 100, 200 and 400 nm, the length sweeps a range from 10 to 2000 μm , and the effective width from 10 to 300 μm .

If the final selection has been based only on behaviour line diagram, a high number of line would have been used. This graph, heavy and unclear, is represented in appendices (page 113). A disappointing observation from this plot is that nearly all the intersections between actuator and film lines are related to displacements under $0.4 \mu\text{m}$. Displacement being evaluated by SEM, this can be problematic to evaluate the smallest displacements (around 10 nm in the global graph), if one reminds that the SEM resolution is about 15 nm [32]. From 3D plots, it is known that higher displacement range cannot be obtained for levels of biaxial strain below 4%, so these curves are kept. Indeed, Raman can be used to determine the strain level if it cannot be done by SEM.

The aim here being to evaluate the impact of strain variation induced by dimensions sweep, only few lines are represented in Figure 3.9 for more clearness. Three specimen lines ($10 \mu\text{m} \times 10 \mu\text{m}$ specimen, with a thickness of 150, 400 and 700 nm) and five actuators lines are pictured. By identifying the discrete strain levels used to construct the lines, one can associate the precise level of strain reached by using a particular actuator with this specimen. This graphs permits to predict theoretically that (intersection 3 considered), to reach a strain level of approximately 0.017 in each direction, one can use the combination

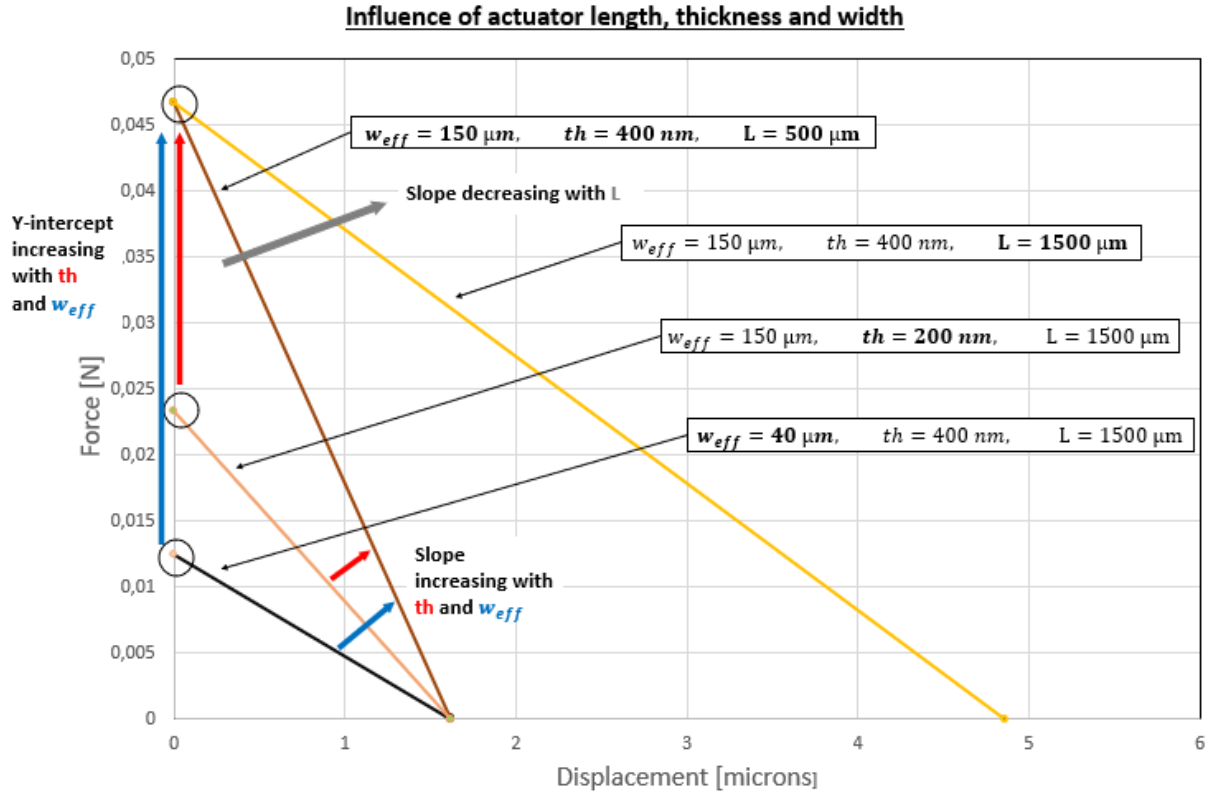


Figure 3.8: Influence of dimensions on actuator behaviour lines (length influence is represented by grey arrow, width by blue arrows and thickness by red arrows). Each line is associated to a certain set of dimensions. The line associated to the 150 μm effective width, 400 nm-thickness and 500 μm -length is taken as reference for influence investigation

of a specimen of 10 μm long and 150 nm thickness and a trapezoidal actuators with effective width expression with $w_{eff} = 40\mu\text{m}$, a thickness of 400 nm and a length of 100 μm . The displacement of the structure after release will be about 170 nm.

Practically, two approaches can be envisaged to sweep a range of strain. Either by varying the dimensions on the structures on the photolithography masks (**length and width** variation), or by varying the thickness of the actuator layer during the deposition process. Depositing many different silicon nitride layer thicknesses being time consuming and requiring some machinery calibration to get the right thicknesses, the first option appears to be more simple. However, choosing 3 or 4 actuator thicknesses could be feasible if needed.

By reminding that strain levels of interest are from 0.001 to 0.04, actuator lines with different values of Y-axis intersect have to be chosen (so selecting actuators with different large basis widths). Taking the three actuator lines with Y-axis intersect at 0.0125 Newton is a good example : they only differ by length ⁵. By just varying the length, a strain

⁵the clear blue line is the shortest possible actuator associated to the effective width of 40 μm without having actuators overlap

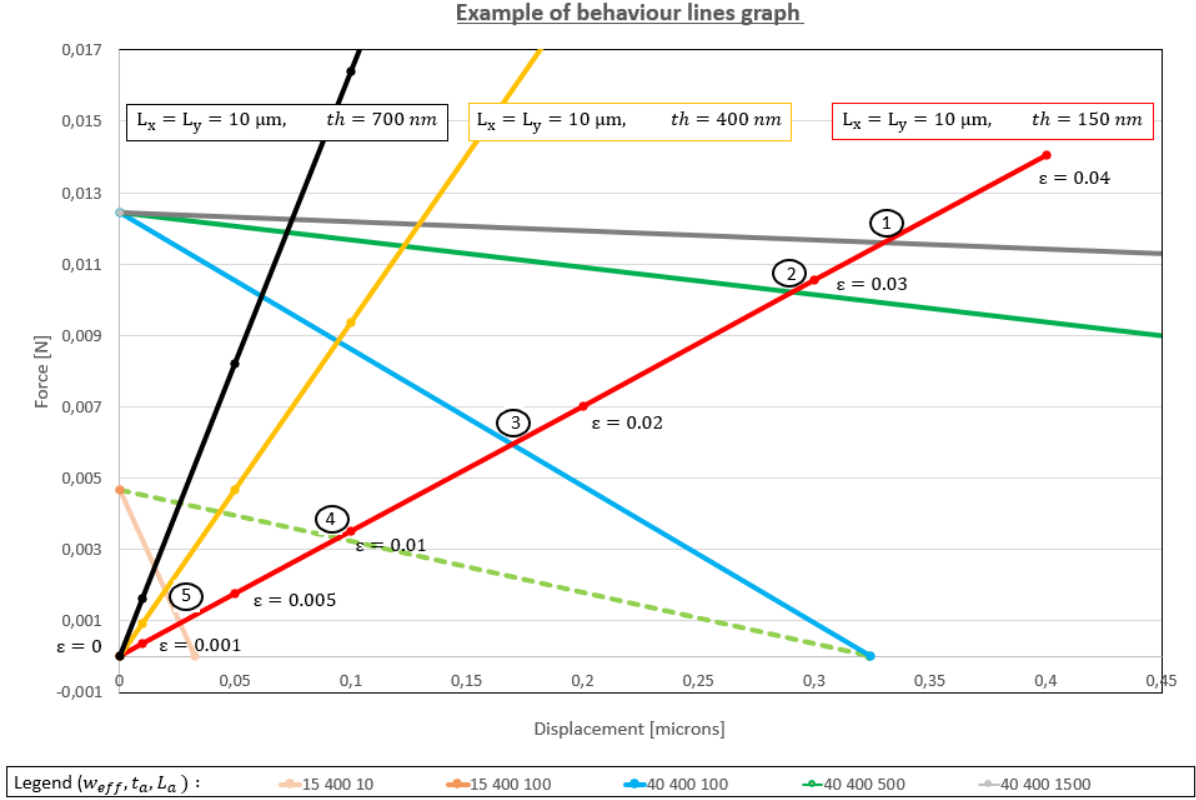


Figure 3.9: Behavior line graph restricted to 8 lines. Lines with a positive slope are associated to the silicon thin films, lines with negative slope to actuators. Each line is associated to a set of dimensions of the component.

range from 0.017 to 0.032 can be reached (intersection 1,2 and 3). It covers a part of the investigated strain range, but not totally. Strains obtained by doubling the actuator length (from intersection 1 to 2) remains quite close, and become even closer when the actuator length increases. If one reduces the effective width to $15 \mu\text{m}$ by keeping length at $500 \mu\text{m}$ and thickness at 400 nm , a strain of 0.009 can be reached (intersection 4), and by reducing the length to $10 \mu\text{m}$ a strain around 0.002 is obtained (intersection 5). Sweeping a wide range of width by keeping other parameters constant generally permits to wipe a broad range of strain. A variation of thickness produces similar effect than width, but for fabrication reasons it is much easier to vary width than thickness. This investigations permits to choose arbitrarily a $10 \mu\text{m} \times 10 \mu\text{m}$, 650 nm -thick⁶ silicon specimen as dimensions to perform finite element analysis. Actuators are 200 nm long, 400 nm thick with small and large basis of $90 \mu\text{m}$.

Performing some length and width sweeps permits to reach a quite broad range of values : the variation of thickness is not essential. This permits to extract the take home message of this investigation, in agreement with the 3D-plot representation : **Coupling the tuning of actuator width and length is the more effective way to reach**

⁶650 nm is the expected thickness of specimen at the end of the process, based on the top-Si thickness of SOI wafers available in WINFAB

quite different strain levels : width variation induces a strong strain variation whereas tuning its length induces a slighter strain modification.

3.2 Specimen design through finite element analysis

Analytical investigation permits to point out the influence of components dimensions on the strain. However, an underlying hypothesis of the development was to consider the strain and stress distribution as uniform in the film, which is obviously not true if we refer to investigations performed on metallic thin film biaxial tensile test [33], [34], [35], [36], [37]. Those papers underline the need to give a convenient shape to the specimen in order to avoid the presence of stress concentration points, called *hotspot*. In the same scope here, one has to go beyond simple analytical model and use finite elements simulations, to deduce one or several specimen shapes considered as convenient⁷ for the present thin film biaxial on-chip microtensile test. The selected FE software is *COMSOL Multiphysics*.

In a first time, FE simulations are dedicated to the silicon film design in order to reach highest possible level of stress before rupture. Then, FE will be briefly used to confirm the trends pointed out by analytical development dimensions range allowing to sweep a certain number of strain levels present in the silicon thin film. In this aim, in addition to sweep a range of components dimensions (like in the analytical development), one can perform some thinning of the center part of the silicon film, called *gauge region*.

3.2.1 Design of convenient thin film silicon specimens for biaxial tensile test

This section is built as follows. After discussing main differences between the present FE simulations and the ones commonly found in papers, the convenience criterion is defined. Then, generalities and parameters selected for investigations are discussed. A simplified cruciform 2D structure is presented, firstly without any improvement so completely inconvenient. In order to get the most suited designs, several structural elements (used in macroscopic metallic thin sheets biaxial tensile test specimens) are included in the aim to meet specifications, like rounded regions or fillet disks at the corner of the central square, slits in the arm to decrease the shear in the central region... Some of them will be selected afterwards to be built in cleanroom. The first presented designs is composed of a cruciform silicon thin film linked to 4 actuators. An alternative design owning only 2 actuators (and presenting interesting advantages concerning SEM measurements) is discussed.

Design of convenient biaxial tensile testing specimens was already investigated for macroscopic/microscopic metal sheets. The challenge here is to start from strategies developed in existing papers for biaxial tensile test macro specimens and to get designs convenient with micro/nanofabrication techniques. For instance, the photolithography resolution is about 1 micrometer, which can be really limiting to build some features of peculiar designs. One has also to pay attention that the amount of matter is quite

⁷Criteria defining this "convenience" will be established further

small and each defect (dislocation, vacancy, impurities...) can be the site of a stress concentration, where failure can occur. Also, materials investigated in publications are macroscopic structures : they are constituted of several grains with different orientations separated by grain boundaries, so that the particular effect of a specific grain orientation is smeared out to get a quite isotropic material. The specimen being made of single crystal silicon, the cubic symmetry of the silicon lattice induces an orientation dependence of mechanical property, such as Young's modulus or fracture toughness (see state of the art). Even if *COMSOL* permits to take this anisotropy into account, the implementation is quite cumbersome so the isotropic hypothesis is still used here. Isotropic and anisotropic simulation runs were briefly performed to check the validity of this hypothesis : it appears that slight changes between the runs occurs concerning hotspots stresses.

In the present case, a design can be considered as convenient if it meets two specifications :

- Having an **homogeneous stress and strain distribution in the center of the specimen**, called *gauge region*
- The level of stress reached in the gauge region is close to the highest one present in the structure, so **the presence of stress concentration at some point outside the gauge region has to be avoided**. The aim is to increase the probability that rupture occurs in the gauge region and no in other places. One can arbitrarily fix that a stress at any hotspot has to be at maximum 3 times the stress in the gauge region.

3.2.2 Global FE parameters and building of the initial system

Generalities and material mechanical coefficient values used in simulations

Solid Mechanics physic from *COMSOL Multiphysics* is used to perform this finite elements analysis. Material physics are based on the 2D linear isotropic elastic material *COMSOL* modelling : this is justified by the plane stress condition and permits to avoid meshing difficulties. In a 2D system, *COMSOL* endows a *Change thickness* option, making possible to set the silicon at a particular thickness and the *SiN* layer with another one. Selected materials are the ones present in the *COMSOL* library : the silicon used is the *n-Silicon (single crystal, lightly doped)* with a Young modulus of 166 GPa and a Poisson ratio of 0.22. The silicon nitride used is the *Si₃N₄ - Silicon nitride* with $E = 250$ GPa and $\nu = 0.23$.

Building the 2D system

The dimensions are those selected at the end of the analytical Excel chart. This particular set of dimensions is considered as a model to identify the stress concentration regions, other structures with different dimensions for the same dimensions ratios will be put on the lithography masks, this is the subject of the second part of this section. The basic system is made of 3 main parts and is represented on [Figure 3.10](#) :

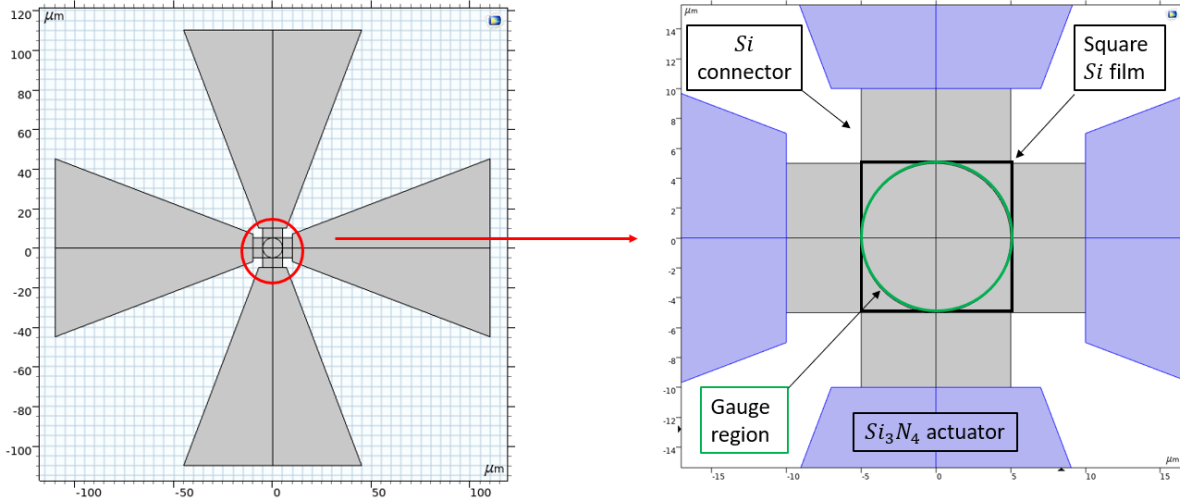


Figure 3.10: (left) : Schematic representation of the initial structure. A focus on the silicon thin film and identification of the different parts

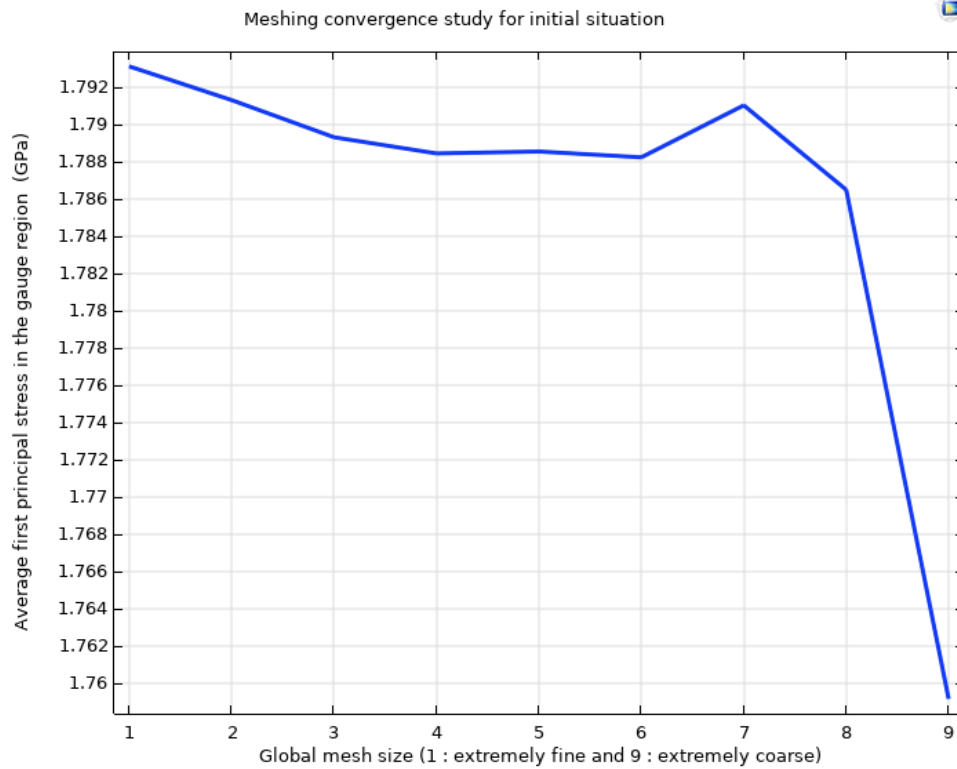
- **the square film** : this film is made of has a edge length of $10\ \mu\text{m}$. A way to reach higher level of strain in the film is to perform a local thinning in the center of the film in a region called gauge region. This allows to increase the ratio between gauge region stress and hotspot stress. It consists of a disk of $5\ \mu\text{m}$ radius centered in the film center⁸. The initial thickness in the square film (inside and outside gauge) is set to 650 nm.
- **the connectors** : 4 rectangular 650 nm-thick silicon connectors make the link between the square part of the silicon film and the actuators. Their length is one half of the film one (so $5\ \mu\text{m}$) and a common width with the film. The complex "film + connectors" constitutes the cross.
- **the actuators** : these trapezoidal actuators are made of silicon nitride. They have a small basis of $14\ \mu\text{m}$ (an override of $2\ \mu\text{m}$ at each ends of the connector), a large basis of $90\ \mu\text{m}$ and a length of $100\ \mu\text{m}$. The override of the small basis is included to ensure a good overlapping between *Si* and *SiN* if slight misalignment occurs during process. The thickness is set to 400 nm. The initial stress present in the nitride is set to 1.1 GPa along $\hat{e}_x$ and $\hat{e}_y$ [32].

Meshing

In order to get mesh independent solutions, a convenient mesh has to be chosen. A triangular mesh with a linear element order is used (one node at each vertice). Convergence study for initial case is available on [Figure 3.11](#). The method used to perform this mesh convergence study is inspired by a *COMSOL* Youtube tutorial [38]. By setting arbitrarily a maximum relative error to 0.2 %, the *finer* seems to be a good compromise between accuracy of results and computation time. To be more accurate, this mesh has the

⁸this circular shape is preferred than a polygonal one to avoid stress concentration near the gauge region corners, see [page 113](#)

following features : a maximum size element set to $8.14 \mu\text{m}$ (almost used in the actuator parts), the minimum one to $0.0275 \mu\text{m}$, a maximum element growth rate of 1.25, a curvature factor of 0.25 and a resolution of narrow regions of 1. Each following design investigation includes a dedicated convergence study to ensure the mesh independence of the results.



Index	Name	Max E.S. [μm]	Min E.S. [μm]	Max E. G.R. [-]	Curvature F. [-]	R. in n. R. [-]
1	Extremely fine	2.2	0.0044	1.1	0.2	1
2	Extra fine	4.4	0.0165	1.2	0.25	1
3	Finer	8.14	0.0275	1.25	0.25	1
4	Fine	11.7	0.066	1.3	0.3	1
5	Normal	14.7	0.066	1.3	0.3	1
6	Coarse	22	0.44	1.4	0.4	1
7	Coarser	28.6	1.32	1.5	0.6	1
8	Extra coarse	44	3.52	1.8	0.8	1
9	Extremely coarse	72.6	11	2	1	0.9

Figure 3.11: Meshing parameters used in the investigated meshes in ??. "Max. E.S." refers to maximum element size, "Min E.S." to minimum element size, "Max E. G.R." to maximum element growth rate, "Curvature F." to curvature factor and "R. in n. R." to resolution in narrow regions

Choice of relevant stresses and strains

One has to represent pertinent stress and strain to highlight the presence of stress concentration at which rupture has a high probability to occur.

A huge majority of published papers about biaxial tensile test investigation concerns ductile materials and are interested in yielding phenomenon even more than fracture. This justifies the use of von Mises stress in order to predict yielding of materials under multi-axial solicitations. Here, simulations do not have the same purpose : at the end, the crucial information we are interested in is to locate the places where failure has a high probability to occur, corresponding to the places where stress concentrates, commonly called *hotspots*. The representative stress has thus to be based on a failure criterion. In brittle materials, failure occurs where the tensile stress reaches the ultimate tensile strength of the material. This is the maximum normal stress criterion [39]. The maximum tensile stress in the specimen is given by the **first principal stress**, that is why this particular stress is selected as qualitative and quantitative indicator for the present analysis. Silicon, or any other brittle material, does not undergo plastic deformation : failure occurs immediately after leaving elastic region, due to the lack of mobility of the dislocation. Note that for ductile material, the ultimate shear stress criterion is preferable to use⁹, which can also be represented by first principal stress in the case of plane stress

Following the definition, the three principal directions are the directions such as the stress can be expressed with only diagonal components. Direction and magnitude of the two principal stresses (in the 2D case the third one is zero) are determined by using the Mohr circle (demonstration is given in Appendices [page 115](#)) and the expression of the first principal stress and strains are given in [Equation \(6.6\)](#) and [Equation \(6.7\)](#).

$$\sigma_I = \frac{\sigma_{xx} + \sigma_{yy}}{2} + \sqrt{\frac{\sigma_{xx} - \sigma_{yy}}{2} + \tau_{xy}^2} \quad (3.8)$$

$$\epsilon_I = \frac{\epsilon_{xx} + \epsilon_{yy}}{2} + \sqrt{\frac{\epsilon_{xx} - \epsilon_{yy}}{2} + \left(\frac{\gamma_{xy}}{2}\right)^2} \quad (3.9)$$

In literature, some other stresses are sometimes selected. For instance, Gassenq et al [27] chose to investigate an particular strain combination with only normal strain ($\epsilon = (\epsilon_{xx} + \epsilon_{yy})/2$) as indicator to investigate strain in Germanium thin-film set under biaxial stress. It might be more pertinent to use the first principal stress, because here it is know that some shear strain components γ_{xy} can be present in biaxial structures [37], it will not be taken into account using the Gassenq formalism, which may lead to an incomplete modeling. Some FE investigations chose to use standard deviation of stress to investigate the stress distribution in their thin film structures [33], others decide to look

⁹But why so ? Ductile materials has the ability to undergo, after leaving the elastic regime, a significant plastic deformation before failing. These deformations, according to dislocation theory, are mainly due to the slide of a part of the crystal along some particular planes, called slip planes. To induce the slip of a dislocation in such plane, the resolved shear stress, defined as the shear stress parallel to the slip plane and to Burgers's vector associated to the dislocation, has to be larger than the critical resolved shear stress, associated to the stress which has to be overcome to induce a motion of dislocation [40]. That explains why rupture is produced by shear for ductile material

at the magnitude of stress and strain [27]. The second option is chosen for the present investigation, standard deviation will only be used to characterise the homogeneity in the film. More precisely, the evaluation of stress homogeneity in gauge is based on the ratio between the standard deviation of first principal stress in gauge and the average first principal stress in gauge. This homogeneity parameter is analogous to [33] one (which used von Mises stress instead of first principal stress, due to the metallic nature of its specimens).

Starting design

Local first principal stress and strain is computed for the initial structure (see Figure 3.12). One notices the apparition of hotspots between consecutive connectors, and a small stress increase at the interface corners between actuators and connectors. More quantitatively, the average stress probed in the gauge region is estimated to 1.79 GPa, corresponding approximately to a 1.5% strain state. The stress present in the hotspots (corners between connectors) reaches 6.61 GPa, the stress near connector/actuator interface is evaluated to 3.2 GPa, and the stress probed in the center of connectors has a value of 2.08 GPa.

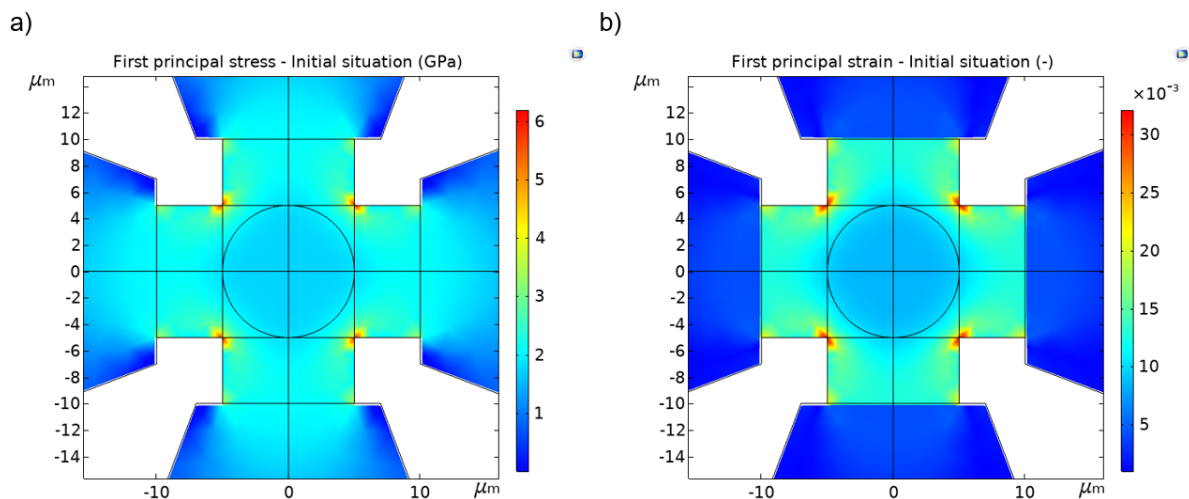


Figure 3.12: Schematic representation of (a) first principal stress and (b) strain for initial situation. Computation was performed by using the *Finer Comsol* mesh. Maximum stress (located at hotspots) reached 6.61 GPa and average stress in gauge region is estimated to 1.79 GPa.

In order to improve the present situation, one has to remind the requirements needed for a convenient design. One of the two specifications is to have homogeneous stress and strain distribution in the gauge region. The standard deviation being evaluate to 0.07 GPa, which represents a variation of 4% compared to the average value. Whereas this specification seems roughly met, the second one, aiming to avoid high stress concentration region outside the gauge region, is more problematic. Indeed, the hotspots stress is considerably higher (3.7 times) than the average gauge center stress. The stress concentration near interface between connector and actuator (1.8 times bigger then the stress in the gauge region) is less concerning because in the real lab on-chip this does not exist, thanks to

the presence of dog-bone specimen extremities. Stress level reached in the arms is lightly above the one in the gauge region (1.15 times larger), which can potentially lead to rupture in the arms instead of in the gauge, as for instance in the results reported by Shimamoto et al. [34]).

3.2.3 Strategy of simulation

In order to retrieve the hotspots and to get a higher ratio between gauge region stress and hotspots maximum stress and between gauge and arm stress, some improvements has to be done. These enhancements are based on several specimen designs elaborated for metallic thin sheets. In the next sections, the focus will be brought on a qualitative improvement first, and then a quantitative analysis will be presented.

3.2.4 Inclusion of rounded corners between connectors

Inspired from [41], [42], [24], [37], [33],[34]. The first investigation aims to smooth the right angle between successive connectors, which constitutes the highest stress level hotspot of the structure . The right angle at the interface between silicon film and actuator is replaced by a $1\text{ }\mu\text{m}$ -radius rounded region. In order to choose a convenient radius R_a for the rounded part, a parametric sweep is performed from $0.05\text{ }\mu\text{m}$ to $4\text{ }\mu\text{m}$ (Figure 3.14). Probes are defined in Figure 3.13 : CC is the rounded domain between connectors, CA in the rounded domain at the connector/actuator interface and the center of connector probe is a point taken at the center of the connector.

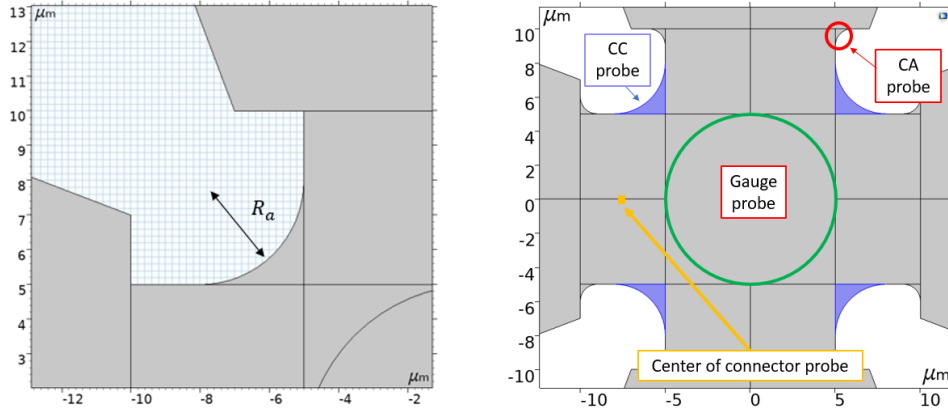


Figure 3.13: Radius of curved area R_a and probed used in investigation

For a very small radius R_a , stress probed near the rounded region increases strongly when radius decreases below $1\text{ }\mu\text{m}$. Maximum stress location is present near the edge of this rounded corner, until R_a reaches nearly $1.5\text{ }\mu\text{m}$ then maximum stress is located near interfaces between connectors and actuators. After this value, this maximum stress increases slowly. One can also notice that stress in gauge regions (and in the center of connectors) slightly decreases as R_a increases, which can be explained by reminding that an increase of R_a is linked to an addition of matter so the force is distributed over a larger cross section. An appropriate value for R_a seems to be located around $3\text{ }\mu\text{m}$: stress

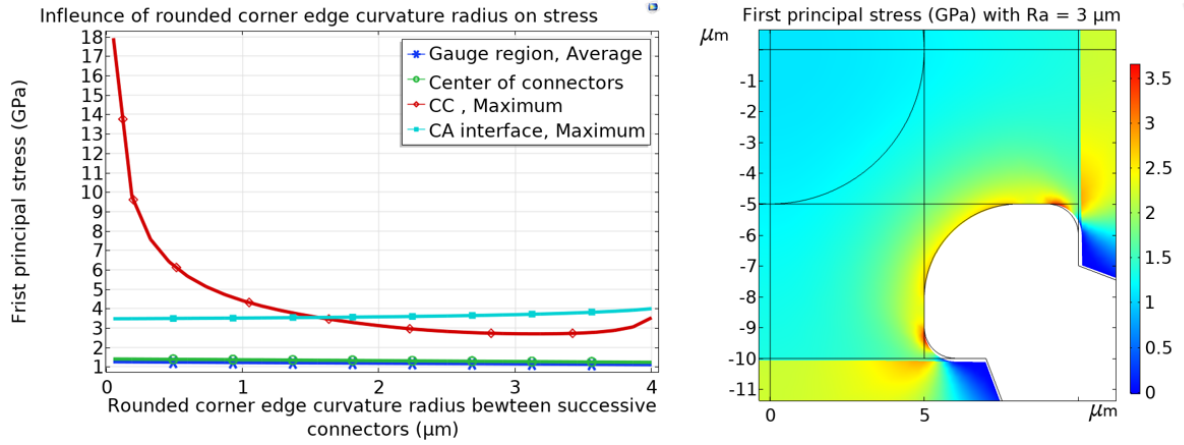


Figure 3.14: Influence of radius R_a of the rounded part between connectors. On right, a schematic representation with $R_a = 3\mu\text{m}$

located in the rounded part is the lowest one, so this value is retained for a $10\mu\text{m} \times 10\mu\text{m}$ square.

3.2.5 Inclusion of filet disks

Inspired from [41], [37], [24], [42], [36], [35], [41]. Instead of including some curvature radius between consecutive connectors, a common practice is to etch some disks in the specimen. This disk is usually centered at the right angle between connectors, or slightly shifted out of the film. Both cases will be investigated here.

Centered filet disk

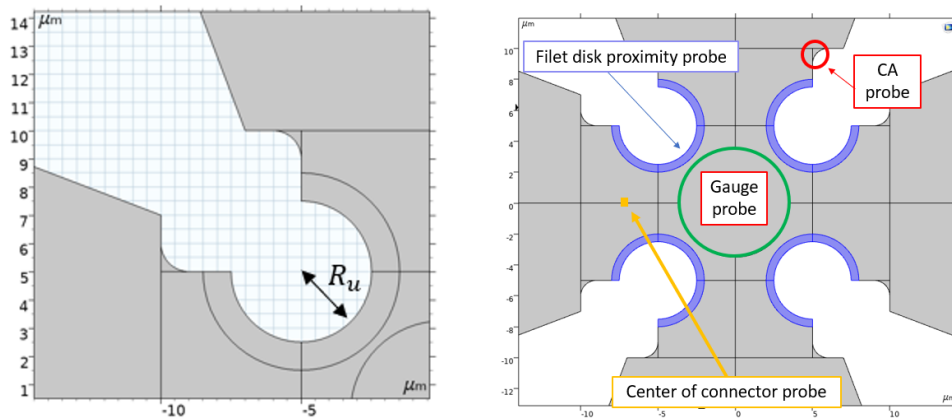


Figure 3.15: Filet disk radius R_u and probed used in investigation

First, centered filet disks are included with radius R_u (see Figure 3.15). A parametric sweep is performed in order to investigate the influence of this radius on the stress level

reached in the different part of the structure (R_u takes values from $0.05 \mu\text{m}$ to $3 \mu\text{m}$). Gauge region radius is reduced to $3.25 \mu\text{m}$ to avoid to induce stress concentration due to file disk in this region. Results are presented in [Figure 3.16](#). Like in the first investigation, a small radius R_u increases the level of stress reached near this region. However, here, the stress probed in the gauge region and in the connectors increases when the radius increases. Indeed, the larger the etched radius is, the smaller is the amount of matter on which the stress can be shared, so this leads irremediably to an increase of stress. The increase of the radius seems to reduces the stress located near the connector/actuator interface. One has to be careful concerning the choice of file radius : even if the $3 \mu\text{m}$ one seems to be the most suited (highest gauge stress level and hotspot stress value close to the minimum one), all radius above $2.5 \mu\text{m}$ reduce the connector width for more than the half of its initial width, increasing the fracture probability in the connector. So we put arbitrarily the upper convenient limit of R_u to $2.5 \mu\text{m}$ and we consider this value as the best one for this particular design. The presence of file disks seems to perturb the stress homogeneity in the gauge region, even if this area is reduced. Indeed, the standard deviation reaches 11% which is comparatively quite higher than the one obtained for other designs, as it will be seen later.

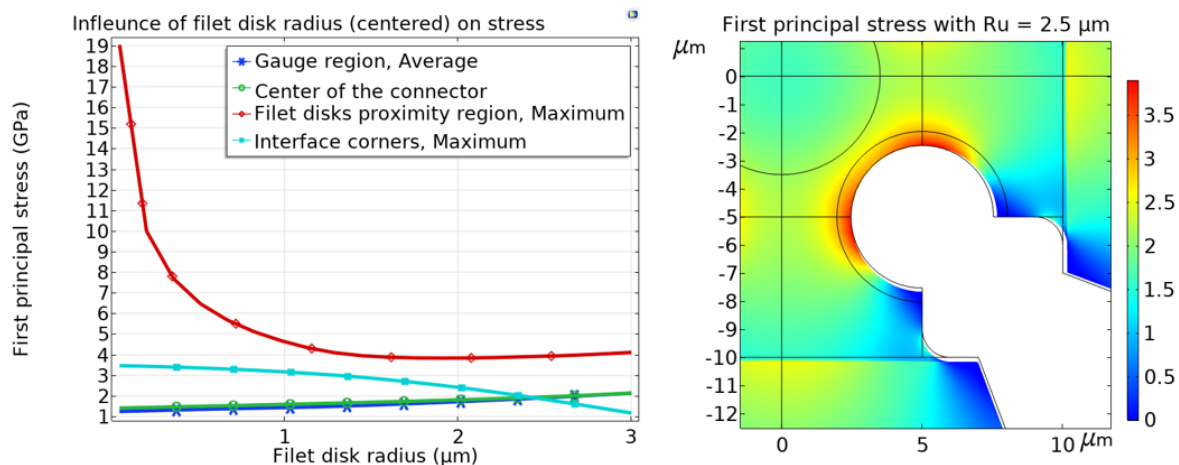


Figure 3.16: Influence of file disks radius R_u (centered on the right angle between consecutive connectors). On right, a file disk of $2.5 \mu\text{m}$ radius is represented.

Shifted file disk

As said before, centered file disks lead to some stress concentration in the connector/square film interface. To avoid this, a common practice is to slightly shift the center of the file disk. Due to the lack a standard value for this position shift, one has decided to arbitrarily take firstly $P = 1 \mu\text{m}$ (corresponding to a shift of $1 \mu\text{m}$ along $\hat{e}_x$ and $\hat{e}_y$). A range of radius R_u from $1 \mu\text{m}$ to $3.5 \mu\text{m}$ is swept and displayed on [Figure 3.17](#). The situation seems enhanced by the shift. Indeed, we can take benefit from the rounded shape by limiting the retrieving of matter. Moreover, stress non-homogeneity problem seems to be solved with this alternative, hotspots being located further. The ratio between hotspot highest stress concentration and gauge average stress is slightly higher (2.3 for shifted center compared to 2.11 for non-shifted) but remains acceptable.

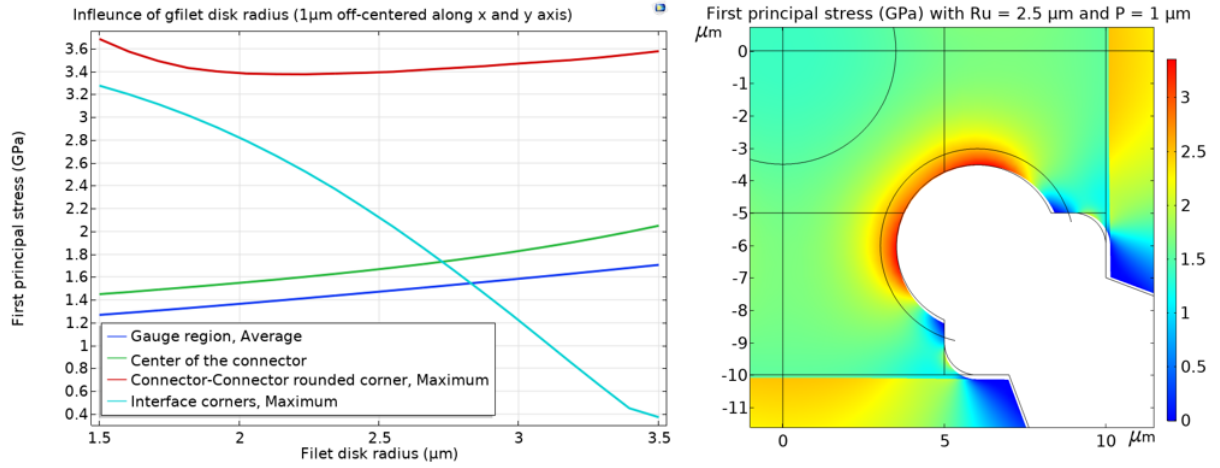


Figure 3.17: Influence of filet disks radius R_u (shifted of $1 \mu\text{m}$ along $\hat{e}_x$ and $\hat{e}_y$). Schematic representation for $R_u = 2.5 \mu\text{m}$ is shown on right

In order to analyse the effect of the excentricity parameter P , one keeps the filet radius R_u to $2.5 \mu\text{m}$ and sweeps a range for P from 0 (centered) to $1.75 \mu\text{m}$ shifted along $\hat{e}_x$ and $\hat{e}_y$. Results are shown in Figure 3.19. Except the stress probed near the actuator/connector interface, first principal stress in places of interest decreases nearly linearly with the same slope. However, even if the stress in the gauge region decreases, shifting filet disks reduces the loss of matter which, as explained before, may limit the premature failure in the connector/square interface. Taking a too large value of P significantly reduces the stress obtained in the gauge region, so choosing a P value of $1 \mu\text{m}$ with $R_u = 2.5 \mu\text{m}$ is a good compromise for a $10 \mu\text{m} \times 10 \mu\text{m}$ specimen.

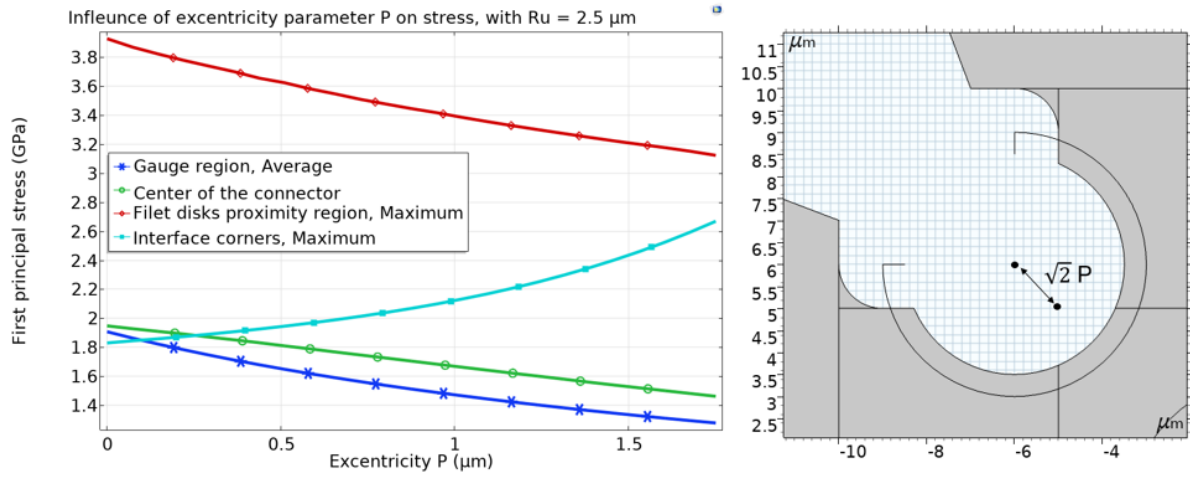


Figure 3.18: Influence excentricity parameter P with $R_u = 2.5 \mu\text{m}$. The definition of P is given by the picture on right

3.2.6 Inclusion of rounded corners at actuator/connector interface

In order to avoid the presence of a hotspot, the right angle formed between connector and actuator has to be transformed by a smoother transition, as usually done for tensile test specimens. A curved transition with a curvature radius R_i is used to replace this right angle. To select a pertinent value of R_i , one evaluates the stress distribution at common places for values of R_i in the range from $0.05 \mu\text{m}$ to $1.5 \mu\text{m}$. Results of investigation are exposed in [Figure 3.19](#). The width of connectors has to be at least $1 \mu\text{m}$ large at each extremity than the actuator ones at the actuator/connector interface to ensure a good overlapping between specimen and actuator even in the case of misalignment during lithography step. For this reason, a radius of $1 \mu\text{m}$ is chosen : the stress near the interface is strongly decreased without causing undesired effects.

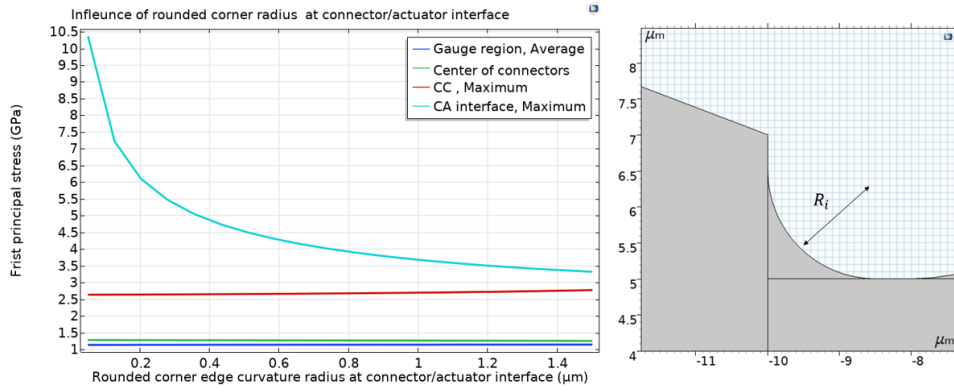


Figure 3.19: Influence rounded corner edge radius R_i near connector/actuator interface.

3.2.7 Inclusion of slits

Inspired from [\[41\]](#), [\[42\]](#), [\[24\]](#), [\[37\]](#), [\[33\]](#),[\[34\]](#),[\[36\]](#),[\[35\]](#),[\[43\]](#). In metallic thin sheet biaxial test specimens, a common method to reduce shear stress in the square part of the specimen, mainly caused by corner effects, is to etch some slits in the connectors in order to reduce the lateral stiffness of the arms, giving so more flexibility to the specimen. To analyse if these slits causes real benefits, one has to check if the stress indicator has to be changed. One chooses to investigate the maximum shear component σ_s , deductible by using Mohr's circle. In the plane stress approximation, one can easily see that the value of this stress one half of the first principal stress value (which thus keeps being a good indicator to investigate the situation improvement).

In many papers [\[37\]](#), [\[35\]](#), these slits have a high aspect ratio in the connector length direction and are really narrow in order to reduce as much as possible the loss of matter. Here, with the photolithography resolution, it is not possible to etch slits with a width smaller than $1 \mu\text{m}$. The number of slits present in the structure is so limited (connectors have a width of $10 \mu\text{m}$). To have slits with a sufficient length, connector length is set to $10 \mu\text{m}$. Indeed, if one want to include more slits or increase their length/width ratio, increasing the dimensions of the connectors is needed (so consequently those of the square

too), and one has to keep in mind that some additional matter is added to the system which could lead to lower applied tensile strain on specimens. Here, shape, length and width of these slits are investigated, in addition to the effect of a slight connector width broadening to hinder the effect of matter removing.

For the slitted structures, a $3\mu\text{m}$ -radius rounded region are include at the right angles between connectors (see first investigation). By constructing the simplest slits system (3 rectangular slits of $1\mu\text{m}$ width and $8\mu\text{m}$ length in each connector), one notices the apparition of hotspots in the neighbourhood of extremity slits (reaching about 5.4 GPa , see appendices [page 117](#) for associated convergence study). Hopefully, from experiments, it is well know that during the fabrication process, the right angle are smoothed thanks to the lithography step. Well, this being known, one can decide to investigate the effect of this curvature effect on the hotspots stress and to deduce the more appropriate curvature. This investigation is the first performed, so that all others use the derived extremity shape in their specific investigations. Investigated parameters are pictures in [Figure 3.20](#)

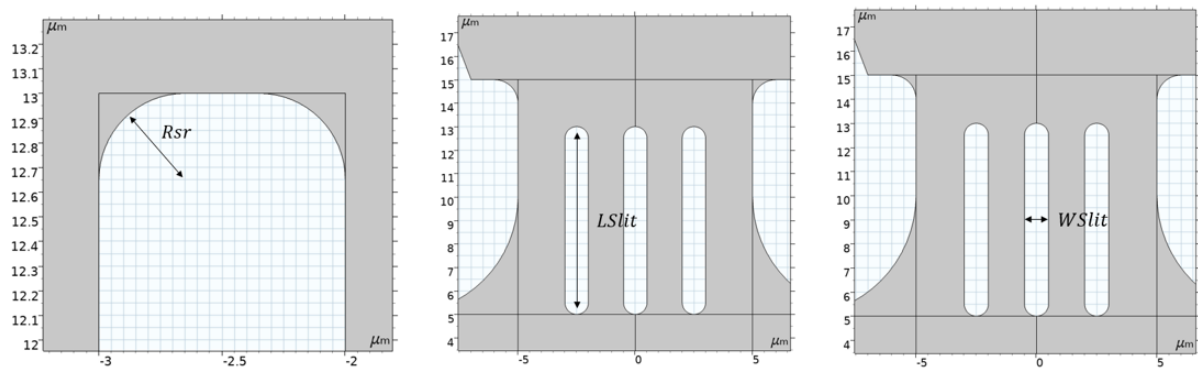


Figure 3.20: Definition of investigated parameters for slitted structure : R_{sr} the radius of slit extremities, L_{slit} the length and W_{slit} the width of slits

Rounded extremities slits

To choose a pertinent curvature radius for slits extremities, a parametric sweep is performed from $0.05\mu\text{m}$ to $0.5\mu\text{m}$ ([Figure 3.21](#)). A $0.5\mu\text{m}$ curvature radius seems to be the best one (leading to the presence of an half-circle at both extremities), this parameter only influences the stress located in the neighborhood the slit extremity (hotspot). The inclusion of these rounded transitions enhances the situation : the ratio between highest stress in the structure and average stress in the gauge region is reduced (like the absolute value of this highest stress). Stress probed at other key points does not seem to significantly change by including these rounded region at the strip extremities. This change permits to reduce hotspot stress from 12.6 GPa to roughly 4 GPa (associated convergence study present in appendices [page 117](#)).

The design with rounded extremities slits is so considered as the "reference" one concerning slitted structures for the further investigations.

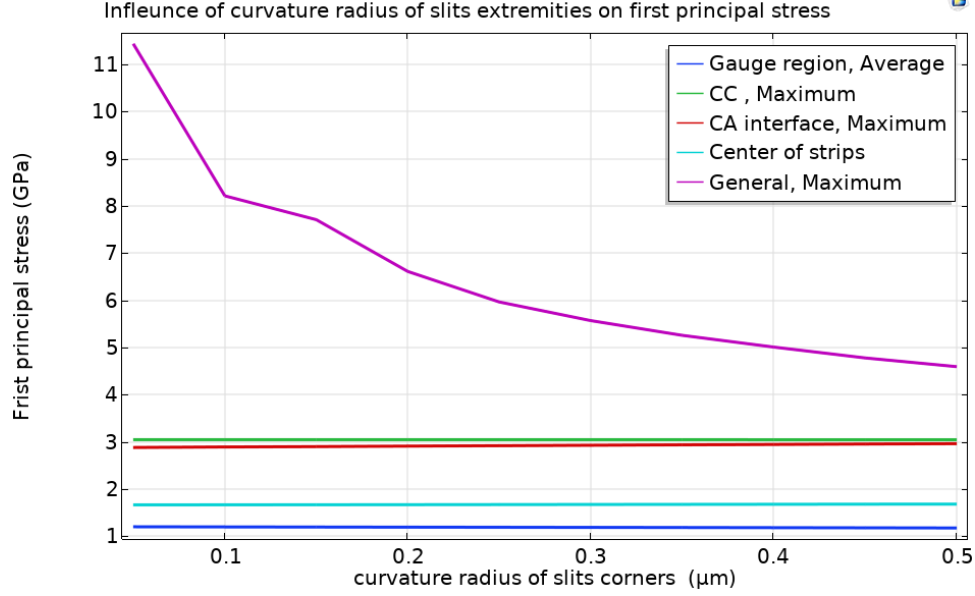


Figure 3.21: Influence of slits extremities curvature radius for a slits with a $8 \mu\text{m}$ length and $1 \mu\text{m}$ width

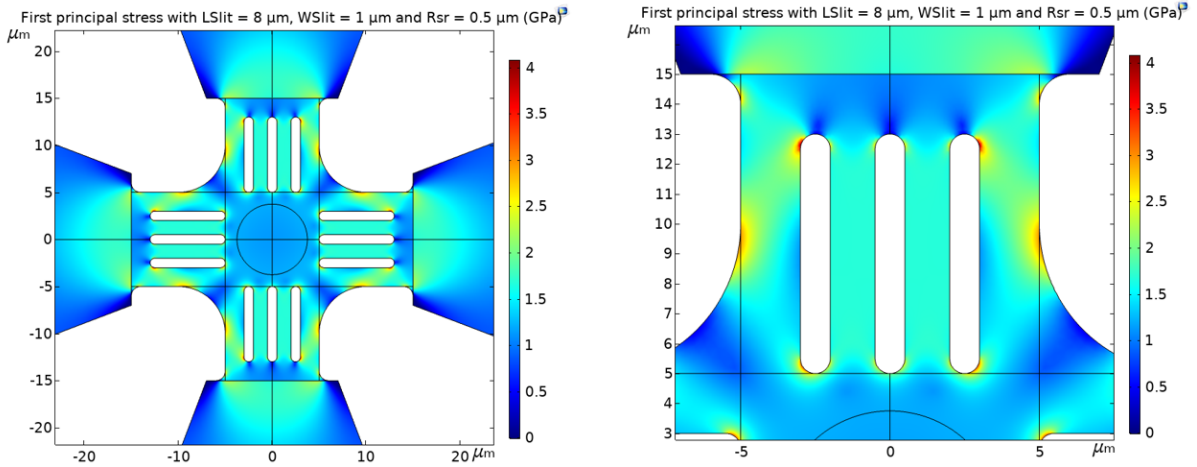


Figure 3.22: Schematic representation of rounded extremities slitted structure with $W\text{Slit} = 1 \mu\text{m}$, $L\text{Slit} = 8 \mu\text{m}$ and $Rsr = 0.5 \mu\text{m}$. One can notice the presence of hotspots near the rounded extremities of external slits

Length of slit

After including rounded transition at slits extremities, the influence of slit length on stress distribution is investigated. From previous paper [37],[43], one expects that slits with a high length/width ratio are the most suited. A parametric sweep is performed firstly on slits length, called $L\text{Slit}$, from 2 to $9 \mu\text{m}$. Results are presented in Figure 3.23. Short slits seem to reduce the maximum stress and to slightly improve the uniformity of the stress distribution in the gauge region (see then standard deviation graph on right). However, the retrieve of matter in the arms produces stress concentration in the strips, the stress being 1.43 times bigger than the average one in the gauge region. In order to

tackle this problem, one can think to add some matter at the extremity of the connectors so that force can be shared in a larger cross-section. An increase of the slits length causes a slight increase of the stress in the gauge region. The maximum shear stress has its maximum for a slit length around $6 \mu\text{m}$, then decreases. If we look closer the order of magnitude of standard deviation change, the change remains quite small so choosing a slit length of $8 \mu\text{m}$ is convenient.

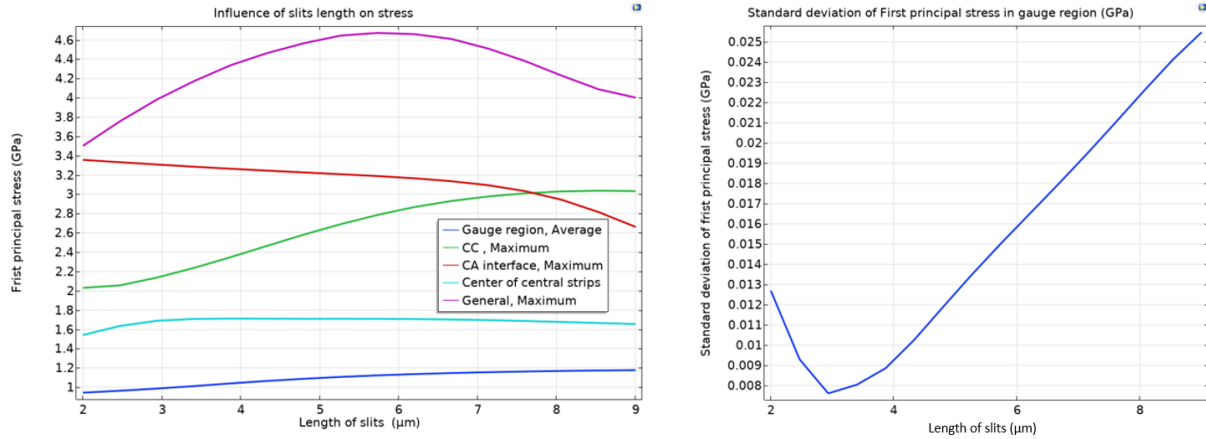


Figure 3.23: Influence of slits length on stress at different probes location. Evolution of first principal stress standard deviation is exposed on right.

Width of slits

The evolution of the stress as a function of the slits width is shown in [Figure 3.24](#). Maximal stress increases with the slit width, whereas stress in gauge region decreases. Even though stress homogeneity is the worst for $W_{Slit} = 1 \mu\text{m}$, this remains better than the one without inclusion of slits. Value below $1 \mu\text{m}$ were computed only to get an insight in the behaviour of structures with high length/width ratio slits. $1 \mu\text{m}$ -width slits are so selected. Indeed, except the slight improvement on the homogeneity, larger slits does not bring any benefit and causes quite higher stress concentration in the strips.

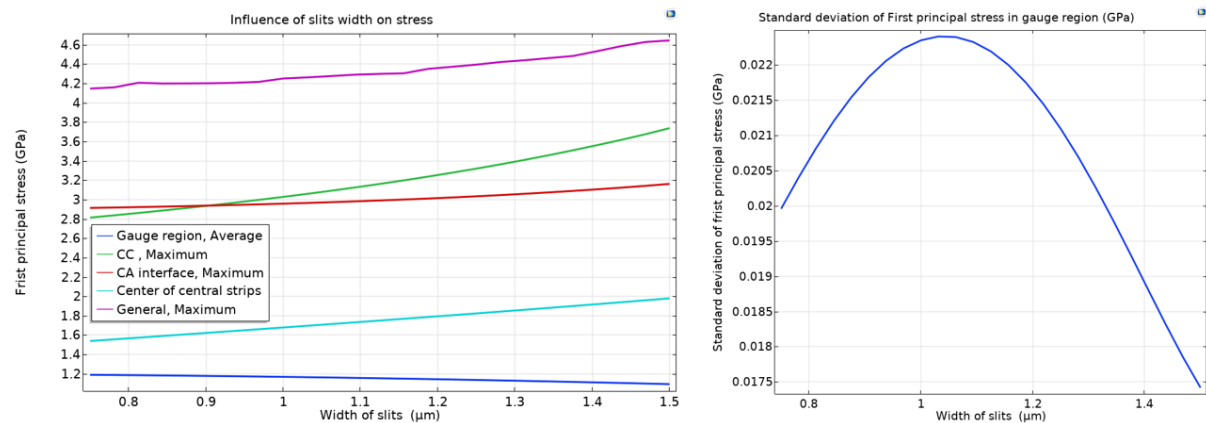


Figure 3.24: Influence of slits width at different probe location

Shape of slits

Until now, rounded-ended rectangular slits have been considered. Another idea could be to use some ellipsoidal slits. Figure of such slits is presented in appendices (page [page 119](#)). It appears that the stress located near the square is bigger than the one obtained with a rounded rectangular one. Design with $1\text{ }\mu\text{m}$ small diameter and $8\text{ }\mu\text{m}$ large diameter ellipsoidal slits is retained for comparison.

Addition of matter

The removal of matter in the connectors induces a stress concentration in the strips. To solve this problem, one can decide to increase the connector width. This is done here by varying the value of the curvature radius. In order to keep the small basis actuator length bigger than the connector width, the actuator small basis is set to $16\text{ }\mu\text{m}$ instead of $14\text{ }\mu\text{m}$ (that explains why the stress level reach for a $10\text{ }\mu\text{m}$ connector width is a bit higher than the one computed before). The curvature radius between successive connectors is set to $5\text{ }\mu\text{m}$ (as investigated before) and a parametric sweep is performed for connector width from 10 to $12\text{ }\mu\text{m}$. Results are shown in [Figure 3.25](#). Situation seems clearly to be enhanced by broadening the actuator width of $1.8\text{ }\mu\text{m}$: in addition to decrease the stress in the strips, the stress located at the hotspots decreases and the stress in the gauge region increases. Around $14\text{ }\mu\text{m}$, stress in gauge region and stress at the center of slits become the same. However, the stress homogeneity in the gauge region seems to be decreased. The connector width of 11.8 is so kept for the final comparison.

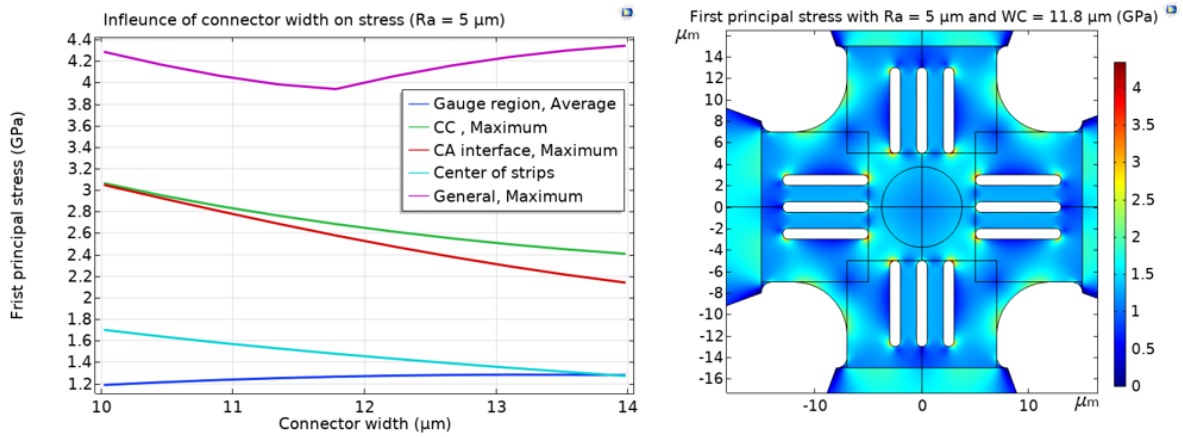


Figure 3.25: Influence of connector width on stress with $R_a = 5\text{ }\mu\text{m}$, $L_{Slit} = 8\text{ }\mu\text{m}$ and $W_{Slit} = 1\text{ }\mu\text{m}$.

3.2.8 Alternative design

Previous lab-on chips dedicated to put silicon micro-beams under strain [\[7\]](#) aim to deduce the strain level of the structure in two ways : Raman measurements and SEM (scanning electron microscopy) measurements. SEM measurements are possible by including some paired cursors : one cursor pair is placed on the unreleased *SiN* frame, the other pair near the interface between the *Si* beam and the actuator, such that during the

release one of the two cursors pair undergoes a displacement. SEM is used to measure the displacement and to relate it to the strain. Unfortunately, the release of the present structure makes the inclusion of cursors not possible : cursors would have to be placed in the neighboring of the actuator/connector interface, on a kind of *SiN* tip (see [Figure 3.26](#)). This tip region will inevitably undergo the release too, so the reference cursors also moves. To solve this problem, an alternative design owning only 2 actuators has been imagined. Only two connectors are connected to an actuator in each direction, the two other ones are attached to the *SiN* frame. Such structure avoids the displacement of the reference cursors during the release. Moreover, even though measurements by SEM with the 4-actuators structures were possible, the reached displacement would be nearly non-measurable by SEM for lowest strain state structures (displacement of a few nanometers) : by having only one big actuator in each direction, the total displacement is obtained is larger. Moreover, they allow more compacity on the photolithography masks than the 4-actuators ones.

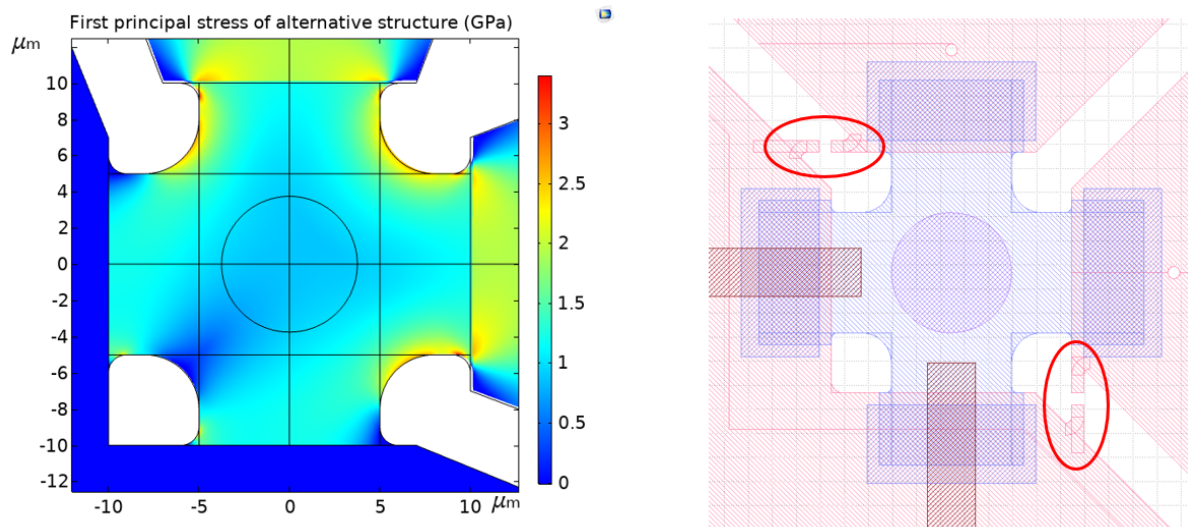


Figure 3.26: First principal stress distribution for the alternative structure

The stress distribution of 2-actuators structure with rounded region is presented in [Figure 3.27](#). The anchoring of two connectors induces some extra shear stress near the connector-connector rounded part, which decreases the homogeneity of the stress distribution in the gauge region (the relative standard deviation is quite increased). Indeed, a tensile region appears on the upper right corner, whereas a nearly zero stress region is present on the bottom left rounded region. The different investigations performed for the 4-actuators structure has also been done for the 2-actuators one, giving quite similar results and trends.

In order to enhance the homogeneity in the gauge region by reducing this shear effect, one can investigate the effect of slits inclusion. Associated convergence study is available in appendices ([page 119](#)). In [Figure 3.28](#), one can notice the disappearance of the tensile hotspot located at the rounded region in the upper right corner, replaced by two more punctual hotspots located at each extremity of this rounded region (the focus on the graph). Stress in gauge region is however reduced compared to the the case without slits.

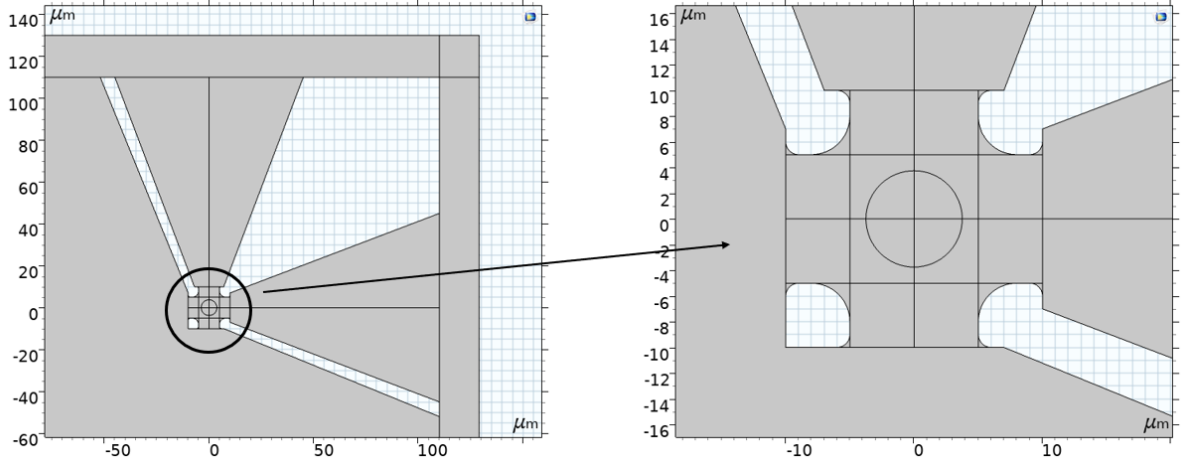


Figure 3.27: Schematic representation of alternative structure. Focus on right

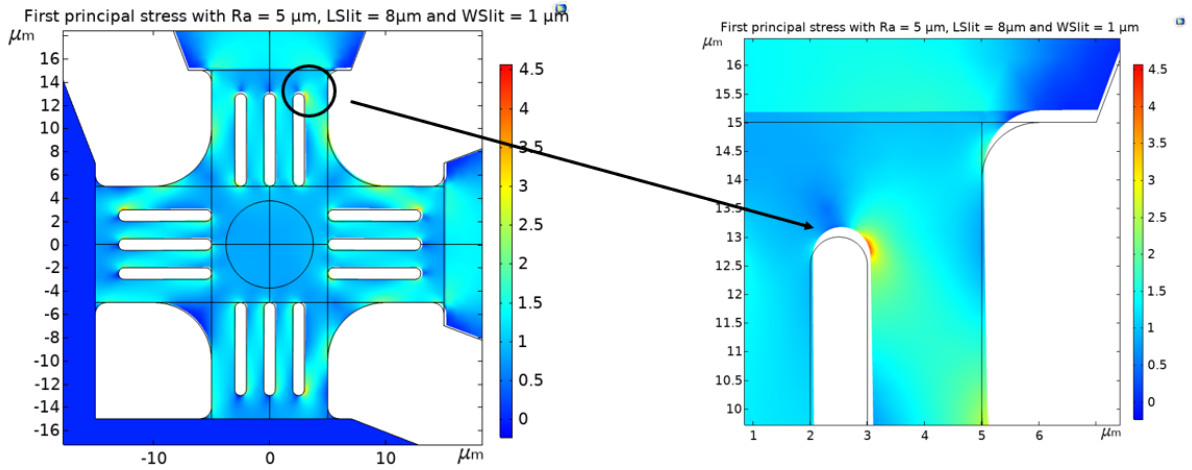


Figure 3.28: First principal stress distribution for the alternative slitted structure. Focus on hotspot is present on right

Like in the basic structure, inclusion of slits causes a stress concentration in the strips. Broadening the connector width might so lead to decrease this concentration. This investigation is presented in [Figure 3.29](#). One can notice that the addition of matter slightly increases the maximum stress in the structure, but reduces quite significantly the stress in the connector. Connector and gauge region stresses for a connector width of $14\ \mu\text{m}$ are nearly equal. This value is retained for the structures comparison.

3.2.9 Effect of local thinning of gauge region

In order to increase the gauge region/hotspot stress ratio in the presented design, one can proceed to a thinning of gauge region ([42],[37], [33]). This approach also constitutes an alternative to reach different strain level without changing the structures on the mask.

[Figure 3.31](#) shows the evolution of stress in gauge region for different thicknesses in the case of a 4-actuator with rounded region structure. The stress strongly increases

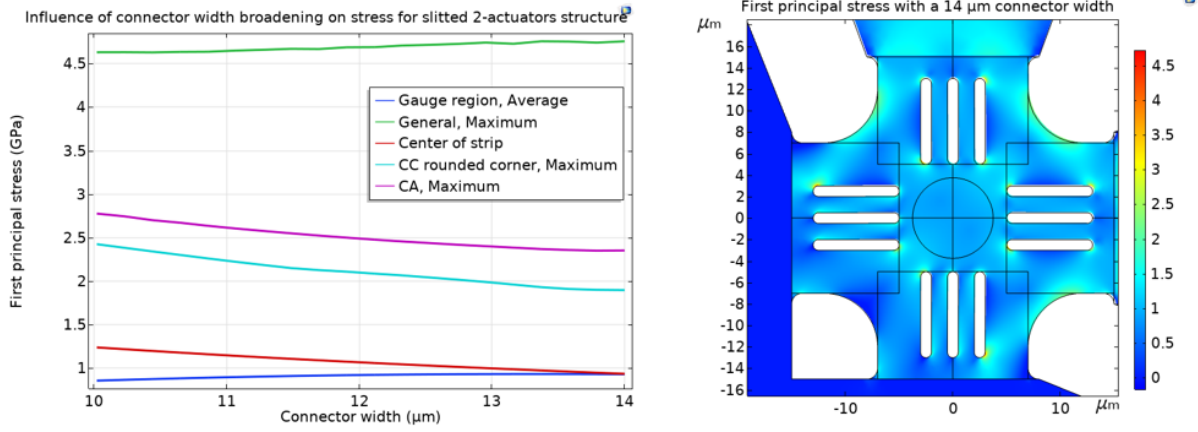


Figure 3.29: Influence of connector width on stress for the alternative slitted structure. Dimensions here are $L_{Slit} = 8 \mu\text{m}$, $W_{Slit} = 1 \mu\text{m}$, $R_a = 5 \mu\text{m}$. First principal stress distribution for a connector width of $14 \mu\text{m}$ is displayed on right.

when the thickness decreases, leading to a larger gauge/hotspot stress ratio. This trend appears for all the investigated designs. The localised hotspots near CA interface migrate to the center of the rounded region edge. Below 550 nm thickness, the gauge region stress becomes larger than the connector one (see scheme B). The ratio between maximum hotspot stress and gauge region decreases as the thinning increases, to shift from 3.36 without thinning to 1.3 with a 50 nm -thick gauge. Moreover, thinning does not impact the homogeneity in the gauge.

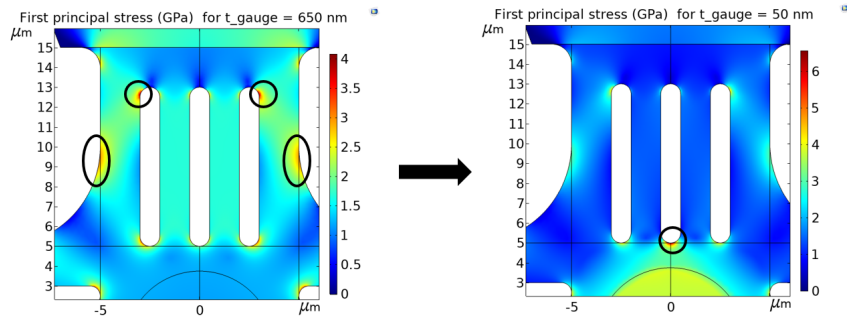


Figure 3.30: Hotspot migration due to the gauge region thinning from 650 nm to 50 nm . Hotspots are indicated by black loops

The thinning has a similar influence for the different inclusions. If one focuses on the hotspots migration, we assist to a displacement from the CA region to the center of the CC edge one for fillet disks, and another particular displacement for the slitted structures depicted in [Figure 3.30](#). Gauge thinning is thus appealing to fulfil the design specifications. As it will be seen, changing this thinning also constitutes a way to tune the strain level in the released structure.

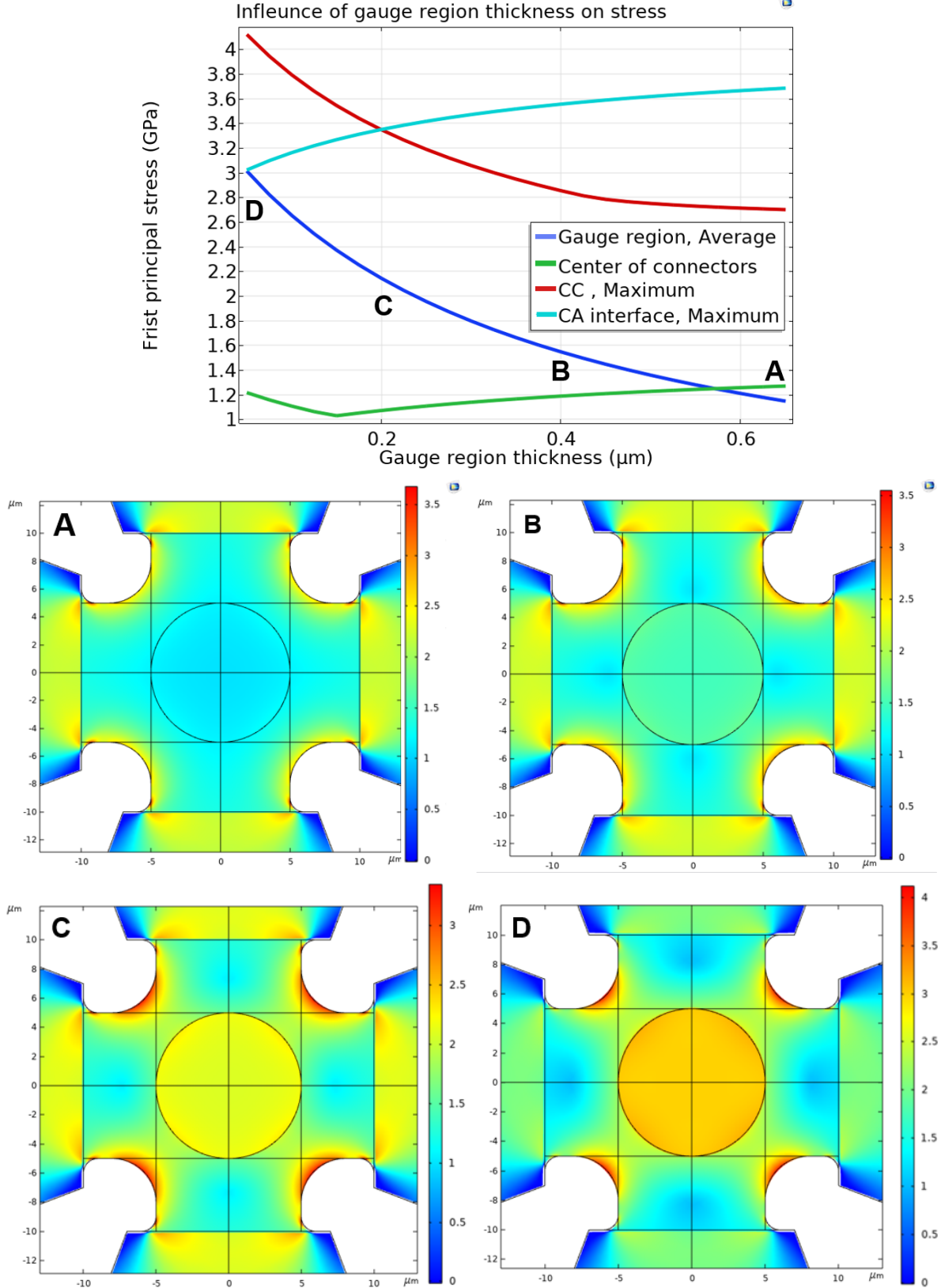


Figure 3.31: Influence of gauge region thickness t_{gauge} on stress with $R_a = 3 \mu\text{m}$. Pictures on right are associated to gauge thickness of 650, 400, 200 and 50 nm and are associated respectively to the letters A, B, C and D on the graph.

3.2.10 Summary of investigations

After having analysed some designs through simulations, the next step is to determine which ones are the most suited. Complete comparative results tables (Table 6.1, Table 6.2 and Table 6.3) are available in appendices (page 119). The dimensions of included elements are those highlighted at the end of each simulation, considered the most suited in each case. In these tables, maximum stress at particular places are resumed : near the actuator/connector interface (shortened in *Interface C/A*), in the rounded region between successive connectors and in the middle of either the connector or the central strip (depending the structure). Average gauge region stress is also indicated, as well as gauge stress standard deviation.

To consider a particular design as eligible, two objectives must be fulfilled : avoiding the presence of stress hotspots while keeping a stress homogeneity in gauge region. Indicators related to these criteria are chosen to be the ratio between the higher hotspot and the average gauge stress, and the ratio between the standard deviation in gauge region and the average gauge stress. More elaborated mathematical tools would certainly give a more accurate informations about this parameter, but the simple use of standard deviation gives a first good approximation (in addition to the fact that a similar homogeneity indicator was used by Demmerle et al. [33]). Table 3.1 displays a ranking of the investigated devices for these two indicators.

Ratio hotspot / gauge stress		Ratio std. dev. (gauge) / average gauge stress	
centered fillet disks (4-actu)	2.11	elliptical slits (4-actu)	1.7%
shifted fillet disks (4-actu)	2.3	rounded extremities slits (4-actu)	1.9%
broadened C width with slits (4-actu)	3.11	rounded CC region (4-actu)	3.4 %
rounded CC region (4-actu)	3.2	shifted fillet disks (4-actu)	3.9 %
rounded extremities slits (4-actu)	3.63	rounded extremities slits (2-actu)	4.5 %
centered fillet disks (2-actu)	3.66	rounded CC region (2-actu)	4.8 %
rounded CC region (2-actu)	3.71	broadened C width with slits (4-actu)	5.5 %
shifted fillet disks (2-actu)	3.83	broadened C width with slits (2-actu)	6.2 %
broadened C width with slits (2-actu)	5.11	centered fillet disks (4-actu)	11%
rounded extremities slits (2-actu)	5.4	shifted fillet disks (2-actu)	16.2 %
elliptical slits (4-actu)	6.03	centered fillet disks (2-actu)	21.4 %

Table 3.1: Quantitative ranking of relative maximum stress (first column) and homogeneity (second column)

As the ratio between hotspots maximum stress and average gauge region stress is concerned, the 4 actuators designs are all better than the 2 ones (except for the design with elliptical slits). The best choice for the 4-actuators designs is the design with included fillet disks. Then follow the structures with circular transition between consecutive actuators and the structures with slits. Concerning designs with two actuators, fillet disks and CC rounded regions seem to be the most suited structures. Slits, however, lead to hotspot/gauge stress ratio above 5. The convenience criterion defined at the beginning of

the section considered a design as eligible if the ratio between hotspot and gauge stress remains smaller than 3. This criterion is only verified for filet disks structures with 4 actuators. However, this ratio is between 3 and 4 for six other designs.

As the homogeneity in gauge region is concerned, the 4 actuators structures remain more suited than the 2 ones. The inclusion of slits had the aim to reduce the shear stress and increase the stress homogeneity. This expectation is confirmed, with the 4 actuators slitted structures (elliptical and rounded extremities ones) present on the top of the ranking, with a relative stress variation lower than 2% in the gauge. For 2 and 4 actuator structures, CC inclusion induces a larger homogeneity than filet disk structures, and especially for the 2 actuators structures. Indeed, filet disks hotspots are located closer than the CC ones, increasing the stress level in the gauge neighbourhood.

3.2.11 Discussion and final choice of specimen designs

Investigated designs present pros and cons. Four-actuators designs meet globally better the requirements than the two-actuators ones. Indeed, the anchoring of two connectors to the fixed frame induces some extra stiffness and impede the film movement in the two directions, creating some tensile area in the opposite corner of the anchored part. However, 4-actuators structures does not allow SEM characterisation, whereas 2-actuators ones do. These two approaches presenting appealing features, it was decided to include both of them on lithography masks.

Whereas some inclusions of element provide good results for the two indicators (structure with rounded region between successive connectors and the structure with shifted filet disks), a trade-off has to be made for some others. For instance, slitted structures appears to be the best suited ones concerning the homogeneity (1.9% of relative standard deviation for the slitted stuctures), but also present a high hotspot/gauge stress ratio.

This master thesis being a version 1.0 for on-chip biaxial loading, a collateral decision was to try several design and not to restrict on the one or two best ones, but to investigate a larger panel of structures. For this reason, slitted structures have also be selected to be put on the photolithography masks. Indeed, even if a whole set of structure came to break, informations like fracture location could be compared to the ones expected by simulation. It could also be interesting to perform a Raman strain mapping on gauge region to confirm the homogeneity enhancement predicted by simulation. The designs put on the photolithography masks are the following ones, pictured in [Figure 3.32](#) :

- 2-actuator rounded region between connectors (Specimen A)
- 2-actuator excentered filet disks (Specimen B)
- 2-actuator slitted connectors (Specimen C)
- 4-actuator rounded region between connectors (Specimen D)
- 4-actuator excentered filet disks (Specimen E)

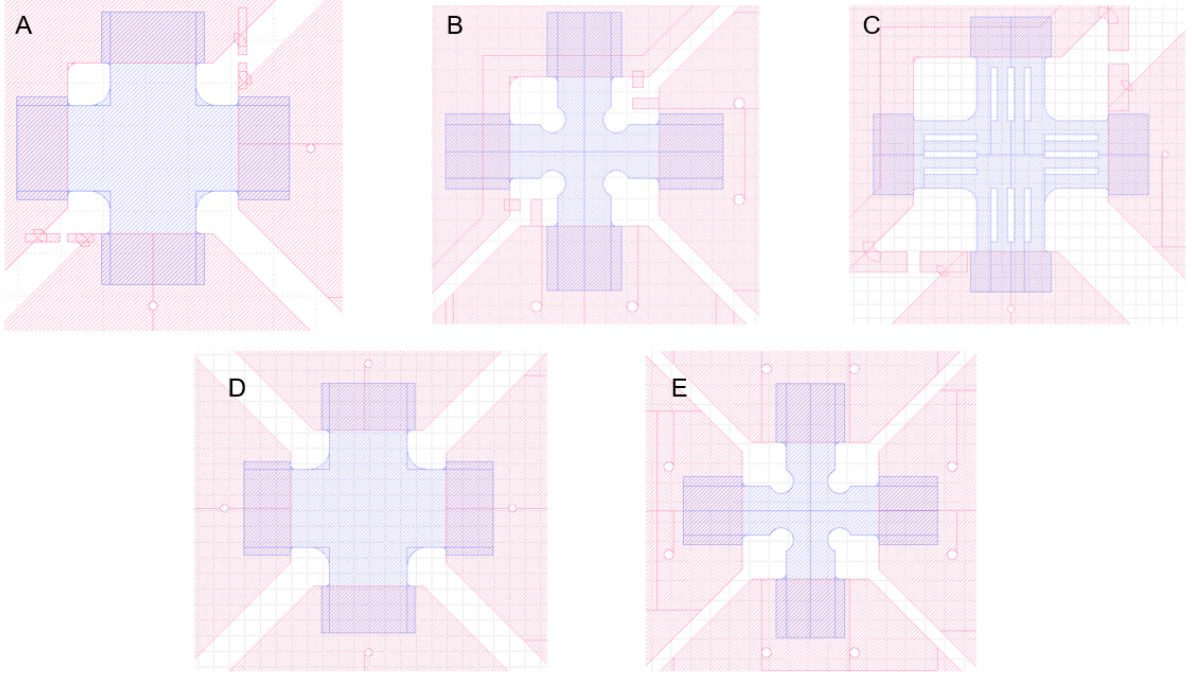


Figure 3.32: Specimens put on the photolithography masks.

3.2.12 Prediction of reached strain levels

Now the specimen are designed, let us focus on the dimensions of the whole system, specimen and actuators. Before focussing on the real choice of dimension, it is interesting to check if the conclusions made for the analytical part are also validated by FE simulations, and evaluate the different of strain obtained with the two methods. Once done, pertinent sets of dimensions are selected to be put on the masks.

From the analytical part, it is expected that changing actuator length permits to sweep a reduced range of strain, and sweeping width allows to reach a wider strain distribution. Let us check if this trend is confirmed by FEA. The 2-actuators specimen with a $10\text{ }\mu\text{m} \times 10\text{ }\mu\text{m}$ central square with $5\text{ }\mu\text{m}$ long connectors with rounded regions between connectors is considered here. Actuator small basis are set to $14\text{ }\mu\text{m}$ ¹⁰. This structure is pictured in [Figure 3.33](#).

Influence of actuator length

The large basis is fixed to $20\text{ }\mu\text{m}$, and the actuator length sweeps values from 100 to $2000\text{ }\mu\text{m}$. Results are shown in [Figure 3.34](#), and are put in parallel with the analytical ones. The two curves present a similar behaviour : a notable increase of the strain for the small lengths followed by a plateau. FEA leads to strains smaller than the ones obtained with the analytical model : the strain level is reduced from 0.0031 (FEA) to

¹⁰in investigations, $12\text{ }\mu\text{m}$ were taken to ensure a $1\text{ }\mu\text{m}$ overlap at each extremity. Taking $14\text{ }\mu\text{m}$ gives even more security in the case of a possible photolithography mask misalignment during the actuator patterning

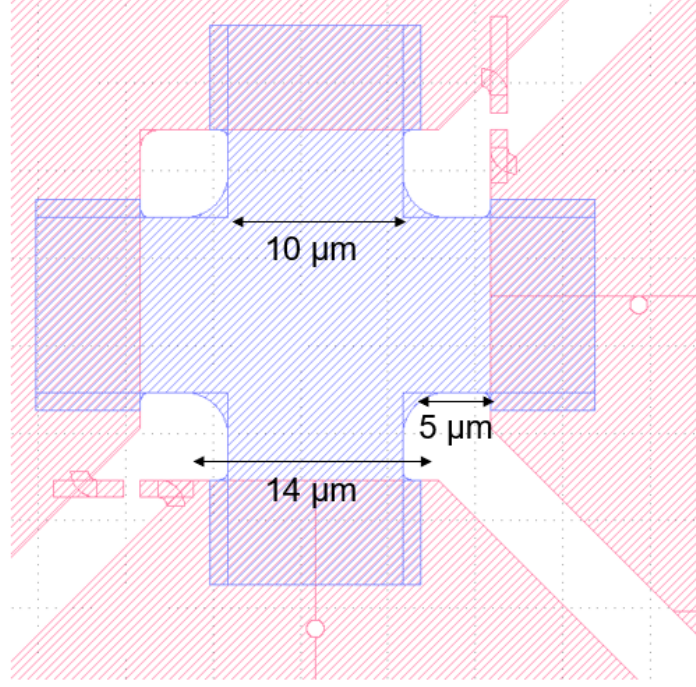


Figure 3.33: Specimen and dimensions considered for the FE dimensioning

0.0024 (analytic) for the shortest actuator and from 0.0034 to 0.0029 for the longest one. This difference originates in the hypothesis set to derive the analytical expression : firstly, the specimen is considered to be a perfect square in analytic, and consists in a circular region in the center of a cross in the simulations. The amount of matter on which the stress distributes is thus different, due to the presence of the connectors and rounded regions. Secondly, analytical development considers a homogeneous strain distribution. It has been seen that, even with improvements, hotspots still exists but are less prominent. A part of the total stress transmitted by the actuators are so absorbed by these hotspots places, reducing the strain reached in the gauge region.

Changing actuator length while keeping width constant permits here to sweep a 0.4 % range of strains. Shchepetov et al. report similar strain variation with its structures, obtaining strains with the same order of magnitude [6]. This confirms the first trend underlined in the discussion of the analytical results : **Changing actuator length gives the possibility to sweep a small strain range.**

Influence of actuator width

Analytical model predicts that a variation of actuator width is far much efficient than actuator length to broad the strain levels. To verify this assumption by FE simulations, a range of actuator large basis from 20 to 1000 μm is taken, for an actuator length of 500 μm . Results are shown in **Figure 3.35** (the width displayed is the effective one). For short actuator widths, analytical and FE analysis give similar strain values (0.028 for FEA and 0.0034 for analytic). When actuators become wider, analytical model provides a higher estimation of strain, presenting a quite linear behaviour between strain and effective width. The strain increases slower for the FE results curve (for an effective

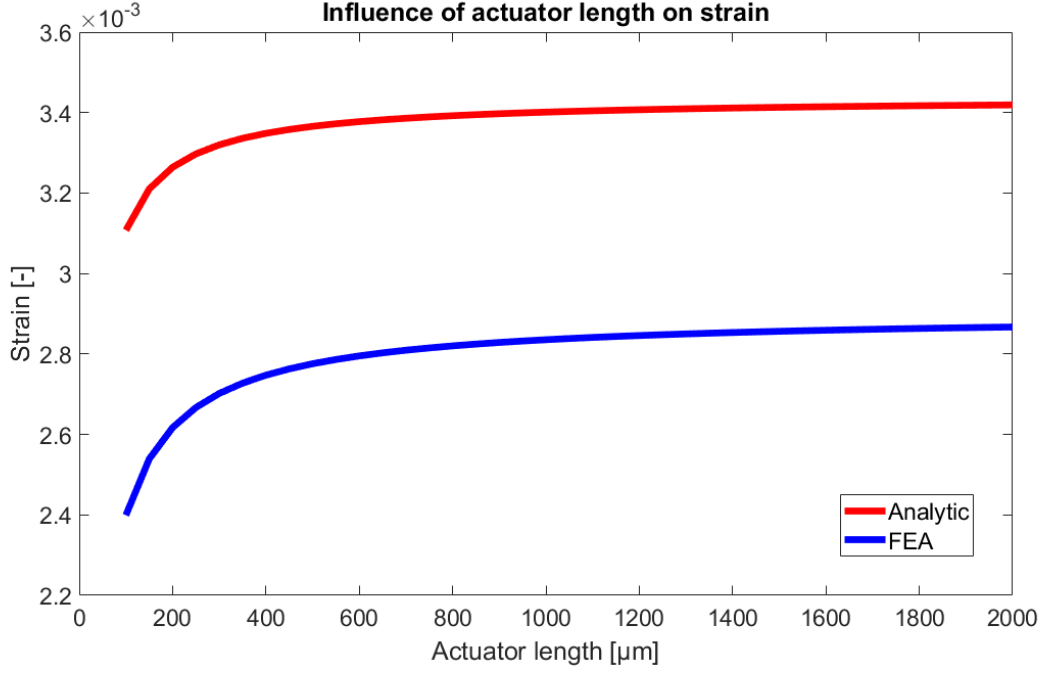


Figure 3.34: Influence of actuator length on strain. The specimen is a $10\text{ }\mu\text{m} \times 10\text{ }\mu\text{m}$, 650 nm -thick with connectors $5\text{ }\mu\text{m}$ long. Actuators are 400 nm thick, a small basis of $14\text{ }\mu\text{m}$ and a large one of $20\text{ }\mu\text{m}$. Blue curve is associated to *COMSOL* results and red one to the analytical model

width of $250\text{ }\mu\text{m}$, 0.022 for FEA and 0.036 for analytic). It can be explained by the increase of stress at the hotspot, which becomes more localised and punctual, when actuator width increases. The gauge stress/hotspot stress ratio decreases, leading to a slower increase of the gauge region strain with actuator width. FEA shows that this variation permits to sweep strain from 0.3 to 2% , confirming the first sentence of this paragraph.

However, tuning only the width for reaching 0.001 to 0.04 strain levels appears to be not optimised. Indeed, this structure is not optimised in terms of compacity : some lengths far smaller than $500\text{ }\mu\text{m}$ permits to reach some lower strain levels. A solution combining both compacity optimisation and strain range maximization consists in **varying both actuator width and length**, and is explained in the following point.

Influence of combined change of actuator length and width

To increase the compacity, the actuator width is based on the actuator length by the following relation : $w_{max}^{act} = 2L_{act} + 14\text{ }\mu\text{m}$. Length is swept from 10 to $1000\text{ }\mu\text{m}$. Results are shown in [Figure 3.36](#). Following *COMSOL* results, taking actuator length from 10 to $1000\text{ }\mu\text{m}$ leads exactly to the desired range : 0.001 to 0.04 . However, the largest structures take about 1 mm^2 in the mask. Having a restricted place on it, one might consider another way to reach value above 2% . A **local thinning of gauge region** is an interesting alternative for this purpose.

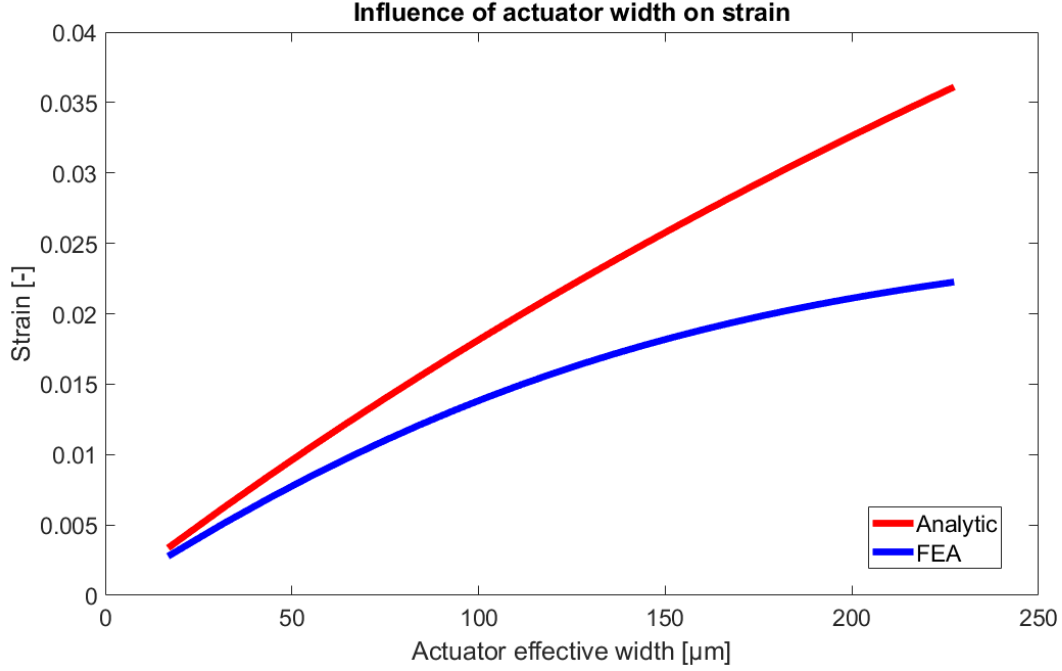


Figure 3.35: Influence of actuator width on strain. The specimen is a $10\text{ }\mu\text{m} \times 10\text{ }\mu\text{m}$, 650 nm -thick with connectors $5\text{ }\mu\text{m}$ long. Actuators are 400 nm thick, a small basis of $14\text{ }\mu\text{m}$ and a length of $500\text{ }\mu\text{m}$. Blue curve is associated to *COMSOL* results and red one to the analytical model

At this stage of the design, the finite etching selectivity between actuator and silicon has to be taken into account. Indeed, the *HF* etches silicon oxide under actuators from their lateral edges. If large actuators are considered, the time needed to release the actuator center can be quite long, damaging the actuator material. To tackle this problem, etching holes can be placed at a precise location to keep acceptable etching time. Minimal spacing between these holes is based on the specimen dimensions. Let us R being the distance etch to fully release the structure (as defined in Figure 3.37. The following relation has to be fulfilled in order to have the specimen released before the actuator :

$$R \geq \sqrt{\left(\frac{L_x}{2}\right)^2 + \left(\frac{L_y}{2}\right)^2} \quad (3.10)$$

Typically, a few microns are added to the right term to ensure the release order. The more efficient way to reduce the etching time is to place them following an equilateral triangles pattern as shown in Figure 3.37. The circles and the line represent the etching front just before the total release of the structure. A progressive release can be obtained with this improvement by varying the etching holes spacing along the actuator length direction, by selecting larger spacing near the fixed frame than near the specimen.

Holes are $1\text{ }\mu\text{m}$ -diameter and put in square. This square has a length of $9\text{ }\mu\text{m}$ for the $5\text{ }\mu\text{m} \times 5\text{ }\mu\text{m}$ specimens and $18\text{ }\mu\text{m}$ for the $10\text{ }\mu\text{m} \times 10\text{ }\mu\text{m}$ specimens. For some structures, like the ones with the rounded region between connectors, the distance to etch to release the film is slightly increased (the total etch front traveling distance is increase of around

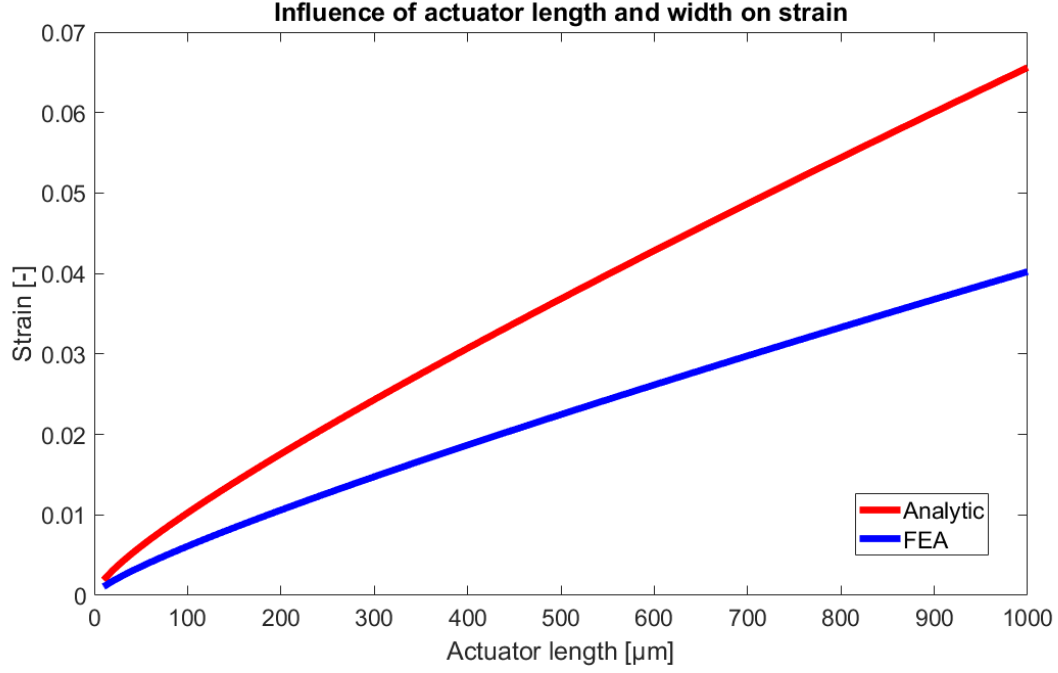


Figure 3.36: Influence of combined actuator length and width on strain. The specimen is a $10\ \mu\text{m} \times 10\ \mu\text{m}$, 650 nm-thick with connectors $5\ \mu\text{m}$ long. Actuators are 400 nm thick, a small basis of $14\ \mu\text{m}$. Actuator large basis is defined by $w_{max}^{act} = 2L_{act} + 14\ \mu\text{m}$. Blue curve is associated to *COMSOL* results and red one to the analytical model

$1\ \mu\text{m}$ for a $2.5\ \mu\text{m}$ rounded radius). This leads to a distance of $4.5\ \mu\text{m}$ to etch for a $5\ \mu\text{m} \times 5\ \mu\text{m}$ specimen. By taking a $9\ \mu\text{m}$ distance between holes, the etch front has to travel a $6.36\ \mu\text{m}$ to induce the actuator release. Theoretically, a $1.86\ \mu\text{m}$ distance has still to be travelled by the etch front when specimen is released, ensuring the first release of specimen¹¹.

Influence of gauge thinning

As said before, thinning the gauge has the consequence to increase the level of strain in the gauge, to increase the gauge/hotspot stress ratio and to modify to location of hotspots. Figure 3.38 shows the influence of reducing gauge thickness or actuator lengths taken from 10 to 600 μm . Whereas the strain is about 0.002 for each thinning value at small actuator length (10 μm), significant differences appear as actuator length is increased, reaching 0.026 for a 650 nm-thick gauge, 0.028 for a 600 nm-thick one, 0.031 for a 500 nm-thick one and finally 0.04 for an gauge thickness of 300 nm. This range of parameters permits to sweep the desired strain range, from 0.001 to 0.04, with an optimized integration degree .

The take home message of this investigation is that **sweeping simultaneously length and width of actuators, coupled with gauge region thinning, allows to reach a broad range of strains while keeping a good integration.**

¹¹This spacing distance is even a bit too large. reducing this spacing leads to a lower actuator damaging. This is one of the most important point to improve if a second version of structure is planned to design.

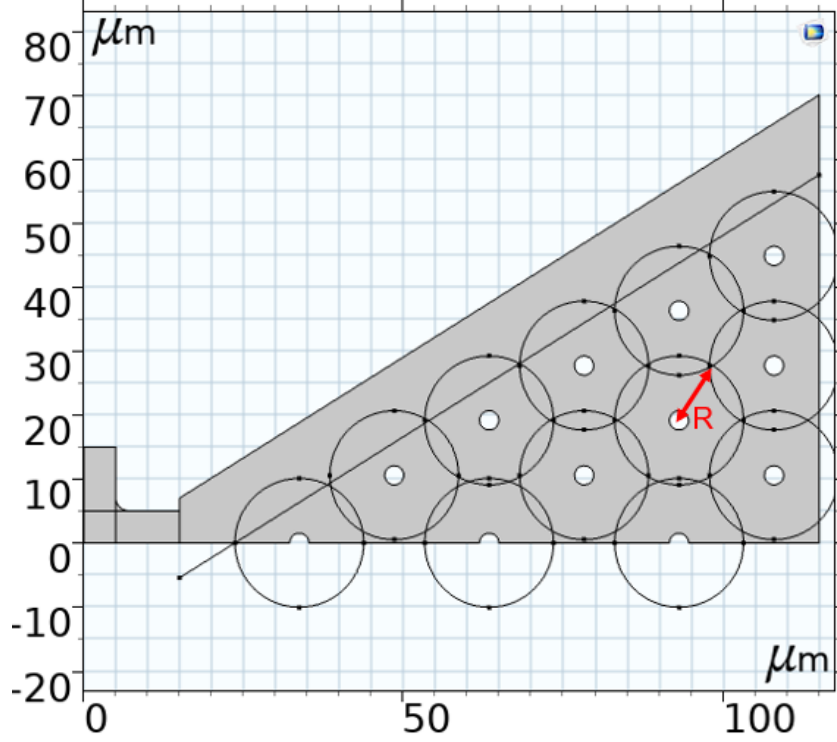


Figure 3.37: Optimised etching hole disposition.

3.3 Choice of dimensions and discussion

Firstly, two sizes of specimens are selected : $5\ \mu\text{m} \times 5\ \mu\text{m}$ ones and $10\ \mu\text{m} \times 10\ \mu\text{m}$ ones. Dimensions ratio is the same for the two sizes.

The effect of components dimensions on strain being known, a series of dimensions can be extracted to sweep strain of interest, from 0.001 to 0.04. The main conclusion of the FE investigation leads to select points on the green curve in [fig. 3.38](#). Etching holes with $1\ \mu\text{m}$ diameter are needed to be include to control the release in those structures. 10 lengths are selected : 10, 20, 30, 50, 100, 150, 200, 300, 400 and $600\ \mu\text{m}$. Actuator large basis is set to $w_{max}^{act} = 2L_{act} + 14\ \mu\text{m}$.

Selecting narrower actuator could also be interesting for several reasons. Firstly, biaxial internal stress in wide structures may lead to their auto-rupture due to the large unmovable anchoring of large basis. Moreover, some papers like Shchepetov one [\[6\]](#), sweep strain ranges comparable to the ones obtained by only varying actuator length. Some interesting comparisons may thus become possible. Thirdly, it is also a way to confirm the trend predicted by FE simulations. Therefore, structures including actuators with $20\ \mu\text{m}$ long large basis with length varying from 100 to $1500\ \mu\text{m}$ are also put on the masks. Wide and narrow actuators structures are represented in [Figure 3.39](#).

[Table 3.2](#) recapitulates all the structures put on the mask. The label *Rounded* in the table refers as rounded region between successive connectors, the rounded radius being

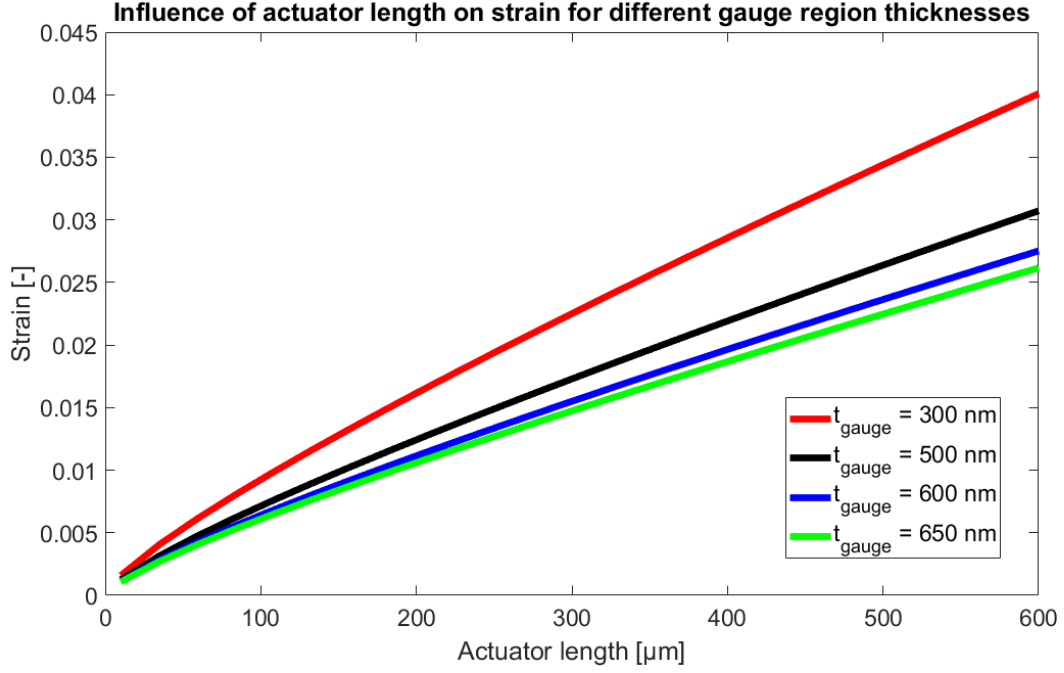


Figure 3.38: Influence of combined actuator length and width on strain for different gauge thicknesses. The specimen is a $10\text{ }\mu\text{m} \times 10\text{ }\mu\text{m}$, 650 nm-thick with connectors $5\text{ }\mu\text{m}$ long. Actuators are 400 nm thick, a small basis of $14\text{ }\mu\text{m}$. Actuator large basis is defined by $w_{max}^{act} = 2L_{act} + 14\mu m$.

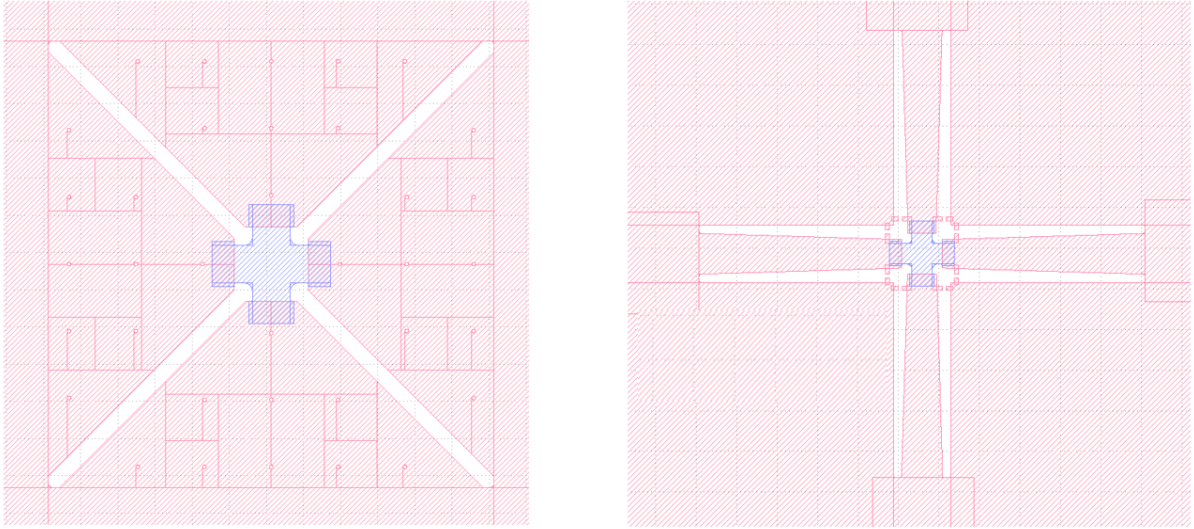


Figure 3.39: Schematic representation of wide actuators structure on left and narrow actuator structure on right

fixed to $2.5\text{ }\mu\text{m}$ for the $10\text{ }\mu\text{m} \times 10\text{ }\mu\text{m}$ specimens (there is hardly no difference with the $3\text{ }\mu\text{m}$ one derived in the FE investigations so both values are well suited). A $1.25\text{ }\mu\text{m}$ one is so selected for the $5\text{ }\mu\text{m} \times 5\text{ }\mu\text{m}$ specimens. As slits are concerned, 3 slits of $1\text{ }\mu\text{m}$ width and $8\text{ }\mu\text{m}$ length are included in each connectors, the central spacing between

slits extremities being set to $1.5 \mu\text{m}$ and the distance between the external slit boundary and the connector edge to $2 \mu\text{m}$. Due to the photolithography resolution, slits are only included in the $10 \mu\text{m} \times 10 \mu\text{m}$ structures. Excentered filet disks structures are present for both $5 \mu\text{m} \times 5 \mu\text{m}$ and $10 \mu\text{m} \times 10 \mu\text{m}$ specimens, the dimensions being those optimized derived previously, namely a filet radius of $2.5 \mu\text{m}$ and an eccentricity of $1 \mu\text{m}$ for the $10 \mu\text{m} \times 10 \mu\text{m}$ specimens, and a filet radius of $1.25 \mu\text{m}$ and an eccentricity of $0.5 \mu\text{m}$ for the $10 \mu\text{m} \times 10 \mu\text{m}$ specimens.

2-Actuators	$5 \mu\text{m} \times 5 \mu\text{m}$	Large actuators	Rounded	thinned unthinned
			Excentered filet disks	thinned unthinned
		Narrow actuators	Rounded	thinned unthinned
			Excentered filet disks	thinned unthinned
	$10 \mu\text{m} \times 10 \mu\text{m}$	Large actuators	Rounded	thinned unthinned
			Slits	thinned unthinned
		Narrow actuators	Rounded	thinned unthinned
4-Actuators	$5 \mu\text{m} \times 5 \mu\text{m}$	Large actuators	Rounded	thinned unthinned
			Excentered filet disks	thinned unthinned
		Narrow actuators	Rounded	thinned unthinned
			Excentered filet disks	thinned unthinned
	$10 \mu\text{m} \times 10 \mu\text{m}$	Large actuators	Rounded	thinned unthinned
		Narrow actuators	Rounded	thinned unthinned

Table 3.2: Recapitulative of structures present on the lithography masks

Throughout this section, some assumptions have been made. The specimen material was considered as isotropic. To increase the accuracy of those simulations, one can take this anisotropy into account by defining an orientation-dependent Young's modulus. Its value can then be evaluated for all the directions based on the compliance tensor coefficients. In this case, one has to orientate the axis system of the sample with the crystallographic orientation of the wafer. Typically in the present case, connectors has to be defined along the $\langle 110 \rangle$. It can be worthy to also investigate the effect of a change of crystallographic orientation.

In addition to consider the specimen anisotropy, one can increase the accuracy of these

simulations by implementing a 3D model. In this way, the overlap between specimen and actuator is correctly represented and the stress distribution near this location can be evaluated more accurately. Moreover, it is useful to represent the effect of tilted flanks surrounding the gauge after thinning on strain, introduced in the chapter devoted to fabrication. However, meshing a flat 3D objects requires additional computational efforts, leading to time consuming simulations.

As it will be seen further, other materials than LPCVD SiN can be considered as actuator. If one attempts to make comparisons between FEA results and experimental ones, the right materials properties have to be given as input parameter simulations. In the case of PECVD SiN, simulations should give quite similar stress distribution, this materials being isotropic like LPCVD SiN. The presence of randomly oriented grains in nickel confers it also anisotropic properties. For nickel, an additional time-dependent simulation could be performed to investigate the creep.

Other designs not investigated here can also be interesting to model. For instance, the square central part can be replaced by a circular one, like in the investigation performed by Shimada et al. [33]. Rectangular connectors can be replaced by trapezoid ones, like did Kelly et al. to subject the part between connector and gauge to a significant deformation perpendicular to the loading direction [33]. Other authors introduced reinforcement structures like rings or plates (also see [33]), but such improvements are not compatible with the existing microfabrication techniques.

Chapter 4

Fabrication process

This chapter is devoted to describe the fabrication process of the on-chip structures, entirely performed within the WINFAB facilities. Section 1 gives a brief reminder about the main fabrication techniques used in the present process. The process used to create the present lab-on chip is presented in section 2. Section 3 summarizes the process in a graphical way. Several optimisation steps have been required, methods and results of the most important ones are summarized in section 4.

4.1 Reminder about micro and nanofabrication techniques

Before tackling the real fabrication process, one has to remind the role and working principle of main fabrication techniques used in *WINFAB* for this master thesis. This includes standard cleaning (not a real process, but useful to detail for process reproducibility), the photolithography, the reactive ion etching, the wet etching, the PECVD thin film deposition, the electron beam physical vapor deposition, the thermal oxidation, the thermal annealing and the critical point drying. Precise operating parameters, often linked to the fabrication machine, are given and justified in section 2. Two characterisation methods used along the fabrication process (ellipsometry and profilometry) are also briefly introduced.

4.1.1 Standard cleaning

The aim of the standard cleaning is to remove organic contaminants and native oxide from the surface of the wafer. It has to be done maximum 2 hours before the next step to ensure the cleanliness of the wafer. The presence of native oxide between deposited layers can have some harmful consequences, especially in a process including an HF release.

The standard cleaning procedure in WINFAB facilities begins by dipping the wafers, placed in a cleaning wafer rack, in a solution containing three fourth of pure sulfuric acid H_2SO_4 and one fourth of hydrogen peroxide H_2O_2 (pirhana solution) heated at 110 °C, for exactly 10 minutes. In this way, the majority of organic contaminants are removed from the surface of the wafers. Wafers are then introduced in a rinsing bath, to undergo

5 fill/empty cycles of DI water. This bath is then filled for 5 minutes with water and nitrogen bubbles are injected. This procedure is repeated a second time but in different bath, aiming at removing the residual organic components which could have survive in the first acid bath, to improve the surface purity. Wafers are then immersed during 15 seconds in an HF 2% solution to remove native oxide, and then immersed in a portable rinsing bath under continuous DI water flow during 5 minutes. Finally, the wafers stand 10 minutes in the "Blue Fountain" with DI water and nitrogen bubbles for the same reason as before, before being put in a rinser dryer, in with the rapid rotation of the wafer rack during a rinsing cycle of 240 seconds followed by a drying cycle of 240 seconds.

4.1.2 Photolithography

Photolithography is a fabrication technique consisting in the creation of a patterned photoresist layer achieving a particular role in a future process step. The surface of the wafer is firstly coated with a polymeric layer, called photoresist, during a phase called *spin-coating*. A photomask, being a glass plate on which chromium has been deposited at some places to form a pattern, is put in contact with the wafer surface¹. A light beam (UV range in *WINFAB*) is then sent through the mask and react with the photoresist coating not covered by the mask. This phase is called *exposure*. Photoresist, once exposed, undergoes a photochemical reaction which modify the local chemical bonds. This bond modification has the consequence to modify the exposed photoresist solubility in a particular solvent, containing typically TMAH (Tetramethylammonium hydroxide). The surface is then immersed with this solvent, and following the type of photoresist, either the exposed or the unexposed part is dissolved and removed from the surface of the wafer. This phase is called the *development*.

In positive photolithography, the photoresist has the property to reduce the polymer chains length under exposure, leading to a greater solubility of exposed resist in the solvent. The opposite happens in the case of negative photolithography (the resist present in the exposed zones has its chemical bonds strengthened). Positive lithography is used to create a mask for an etching, whereas negative one is either used to get the complementary pattern of the mask or to perform lift-off deposition. After these steps, the remaining photoresist is remove during a phase called *stripping* in which the wafer is manually cleaned with acetone then methanol then DI water². The complete scheme of a classic photolithography process is given in [Figure 4.1](#).

Following the aim of the photoresist patterning and the kind of photomask, vertical or tilted photoresist flanks can be desired. Geometry of resist flanks are determined by the exposure dose, consisting in the total amount of light received by unit of photoresist surface. If one aims to perform a lift-off, the photoresist is generally slightly overexposed, whereas if an etching is planned, one aims to get some stiff edges to uniformly achieve the

¹Two kinds of masks exists : the light field ones, with a majority of transparent areas, and the dark field ones, with a majority of opaque zones.

²Acetone is a good, non toxic and low cost solvent for dissolving organic molecules. Methanol has the purpose to remove acetone and the solved organic particles.

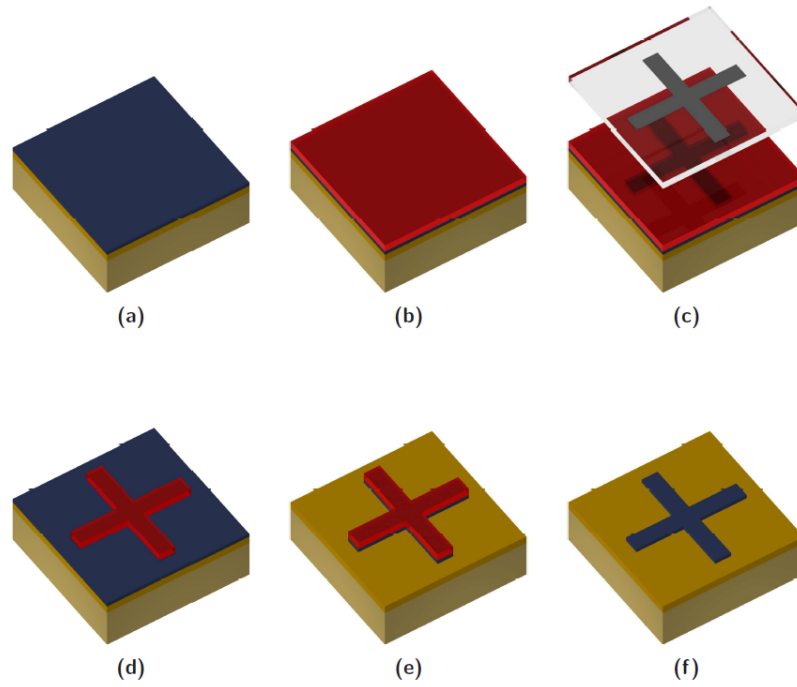


Figure 4.1: Photolithography steps in the case of positive photoresist : (a) initial configuration (a thin film is deposited on the substrate). (b) spin-coating. (c) exposure. (d) development. (e) arbitrary step, like plasma etching of the thin film here, (f) stripping. [22]

masking purpose, even at the extreme edge of the resist.

4.1.3 Etching

The etching in microfabrication consists in removing a complete layer or an unmasked part of a layer by chemical or physical methods to obtain a desired pattern. Two kinds of etching exists in microfabrication : the dry etching and the wet etching. The difference between the two concerns the phase (plasma or liquid) of the etchant. Before presenting them, concepts of etching selectivity and anisotropy need to be introduced.

In order to etch particular areas of a layer, one has to protect the other areas with etching masks. The choice of the mask material is based on the **etching selectivity** between this material and the material to etch, defined as the ratio of etch rate between different species by using a particular etchant. An etching can sometimes be performed on a multi layer structure. If one aims at etching the top layer, a stopping layer has been used if the etch rate of the underlying layer is non-negligible in the etchant. This layer is also chosen based on the selectivity criterion and the compatibility with other process step.

During the etching, the material under the mask can be removed in specific directions to obtain geometric entities such as sharp corners, deep cavities. The etch rate in the different directions are so unequal, the etching is so said anisotropic. Both dry and wet

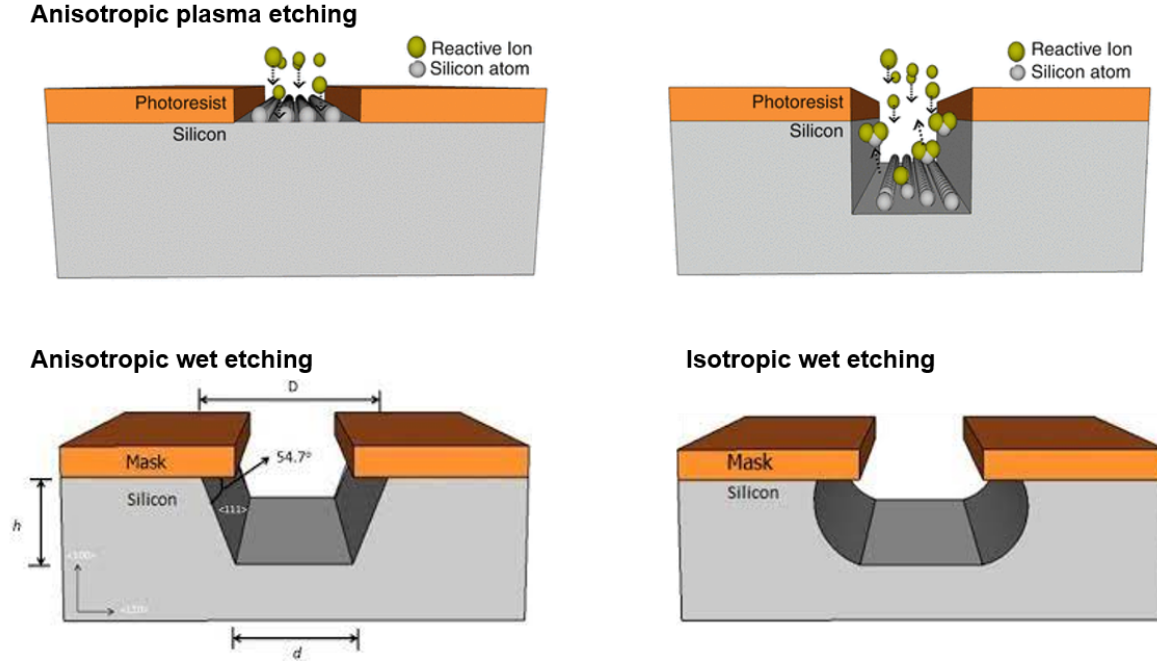


Figure 4.2: Difference of structures obtained after isotropic and anisotropic etching. (up) : anisotropic plasma etching of silicon. (down) : on left, anisotropic wet etching of silicon. The plane $\langle 111 \rangle$ are exposed. (right) : isotropic etching of silicon[44]

etching can be anisotropic, but the physical reason is different. In wet etching, anisotropy emerges from etching rate difference between the different crystallographic directions. An example is the TMAH wet etching of monocrystalline silicon : the presence of a higher number of dangling bonds in some directions induces the creation of flanks along the four $\langle 111 \rangle$ planes. In dry or plasma etching, the surface is bombarded by ions as explained hereafter and an inhibitor is used to protect the side-walls of a cavity during ion bombardment [45]. It is thus possible to obtain structures with high aspect ratios, highly anisotropic. If the etching has the same etch rate in each direction, it is said isotropic : its the case for wet etching of amorphous materials, like the HF etching of SiO_2 or for dry etching of silicon by XeF_2 . The difference between structures obtained by anisotropic and isotropic etching is represented in Figure 4.2.

Reactive ion etching (RIE)

Plasma etching consists in creating a gas plasma in a low pressure chamber and applying a RF frequency to accelerate the ions towards the surface of the wafer (configuration of the chamber is exposed in Figure 4.3, left). These ions bombard and chemically react with the sample surface, transforming the surface atoms into volatile and/or smaller molecules which are then removed by the vacuum system. The orientation of the ions due to the applied electric field (RF frequency) makes this etching quite directional. Etching gases are selected to produce volatile species during reaction with the sample surface. Typical etching gases are halogens, fluorine, chlorine and bromine, produced from SF_6 ,

CF_4 , CHF_3 , C_4F_8 , Cl_2 , BCl_3 , $SiCl_4$ and HBr .

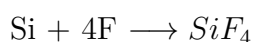
Dry etching performed in this master thesis is a particular kind of RIE called *Inductively plasma coupled - RIE*. In this technique, a high density plasma is created by an inductively plasma source. In opposition to classic RIE, ICP RIE permits to separate the control of the plasma creation, by controlling the ion fluxes, to the 13.56 MHz RF bias applied vertically to extract and accelerate ions to the wafer surface. This permits to decouple the control of the etch rate and the etching anisotropy.

RIE is used in this master thesis to etch silicon and silicon nitride. Both are based on fluorine chemistry.

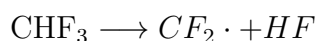
RIE of silicon is independent of crystal planes, so any shape can be fabricated, unlike anisotropic wet etching using TMAH as etchant for instance. In WINFAB, RIE of silicon is performed by injecting a mixture of CHF_3/SF_6 in the chamber. The silicon etching by fluorine chemistry is primarily due to the free fluorine, created by electron impact dissociation reactions of SF_6 of the form (x going from 3 to 6) [46]:



The silicon etching occurs by a reaction with F atoms :



While the primary etchant is provided by SF_6 , CHF_3 provides CF_2 , a precursor for polymerisation reaction which has the aim to protect sidewalls from etching (Figure 4.3, right) :



This permits to etch the unprotected silicon areas in an highly anisotropic way [47].

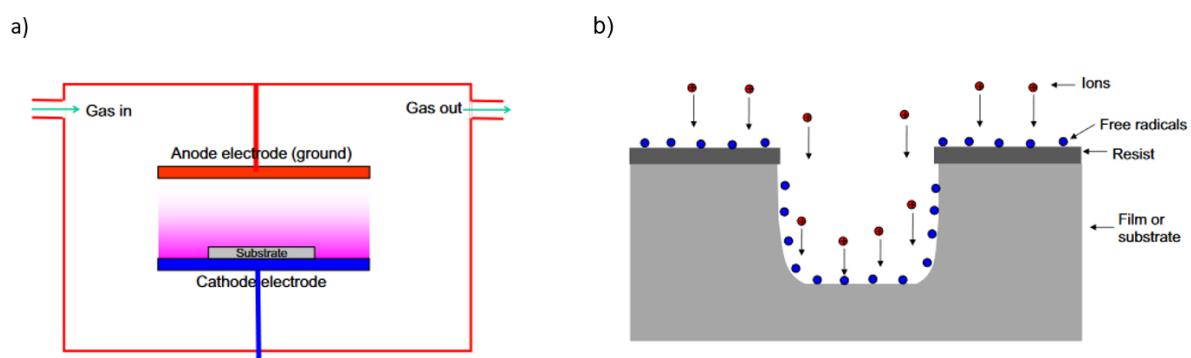


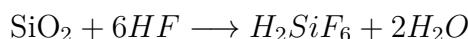
Figure 4.3: Representation of a plasma etching chamber (on left) and the dry etching process occurring at the surface of the sample (on right) [48]

RIE of silicon nitride is performed in WINFAB by using a CHF_3/O_2 plasma. CHF_3 here is responsible of the SiN anisotropic etching by providing fluorine, which reacts with SiN to form volatile species like SiF_x and NF_x [49], and precursor for polymerisation reaction aiming at protecting the sidewalls³. The presence of O_2 in the plasma serves to avoid excessive polymer deposition from the CHF_3 plasma [51], leading to smoother sidewalls.

Wet etching

Wet etching is a purely chemical etching technique, using a liquid etchant. Very inexpensive and simple to implement, this technique is widely used in manufacture of printed circuit board. In opposition to plasma etching, wet etching is most often isotropic, meaning that the film is etched vertically and horizontally at nearly equal rates. A consequence is an undermask etching and a curved etching profile, as shown in [Figure 4.2](#). Wet etching can also be anisotropic in the case of monocrystalline materials, wet etching produces flanks following a particular cristallographic direction, as explained before.

Wet etching is used in the present process firstly to perform the final release (etching the BOX layer with hydrogen fluoride HF), and secondly to ensure the good overlap between the film and the actuator (by using buffered HF solution "*BHF*"). Indeed, the selectivity between SiO_2 and Si in HF is estimated at 1000:1. It was also considered as a possible way to perform the thinning of the specimen gauge region. *HF* etches SiO_2 isotropically following the reaction :



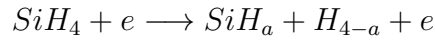
4.1.4 PECVD thin film deposition

PECVD, or plasma enhanced chemical vapor deposition, is a particular CVD technique used to deposit thin films on a substrate. The components of the film are injected in the chamber in gas state, adsorbed by the surface and reach a solid state to constitute the thin film. PECVD can be viewed as a variant of LPCVD in which a plasma is used to decrease the temperature of the substrate at around 300 °C. This low temperature deposition generally give PECVD films a porous microstructure [52]. Reactive species are thus in a plasma state, and ions are directed toward the sample by applying a RF voltage between two parallel plates. A consequence of the energetic ions bombardment is the increase of the thin-film density and a more simple contaminants elimination. The chemical reactions occurring in the presence of a plasma are quite complex, and involve generally bond scrambling, which are often incomplete.

PECVD is used here to deposit silicon nitride thin films, in substitution to LPCVD. Sputtered silicon nitride films also exist, but own a lower level of internal stress. Input gases used in *WINFAB* for PECVD thin film growth are SiH_4 , NH_3 and N_2 . A scheme of the chamber is present in [Figure 4.4](#). The film growth process is achieved by a radical

³complete radical polymerisation can be found in [50]

reaction [53], including the following steps. Firstly, a gas phase dissociation occurs for the two gaseous input species :



Then, a SiN thin film is formed by species insertion : an amorphous Si:H-like layer is formed on the silicon wafer by SiH_a and NH_b radicals. $Si - N$ bonds are then formed through rupture of excessive NH_b (and SiH_a) radicals and replacement of the existing Si-H bonds (and N-H ones) in the film following the reactions :

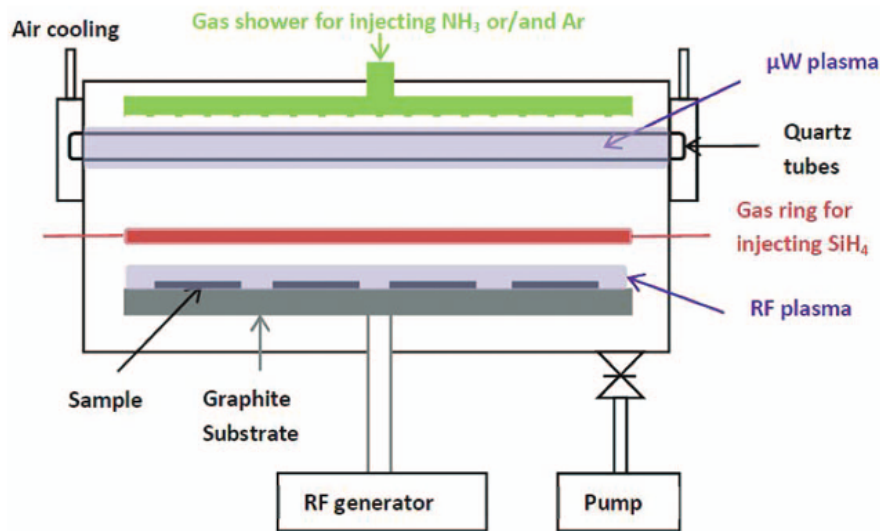


Figure 4.4: Representation of a PECVD chamber, especially dedicated to the deposition of SiN thin film [53]

4.1.5 Thermal annealing

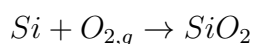
Thermal annealing is common fabrication process used to modify the chemical and physical properties of a structure. Indeed, in MEMS fabrication, some film depositions like *PECVD* occur at quite low temperature. The microstructures obtained after those steps may not be desirable since molecules of the film lack sufficient kinetic energy to reach a thermodynamic equilibrium state. Hopefully, the situation can be enhanced by performing such thermal treatments, providing additional driving forces for film structural reorganisation. Some process requires sometimes high temperature steps after a low temperature deposition. Thermal annealing can be used to permit this reorganisation, leading to a more stable film. Moreover, this thermal process might also generates intrinsic

stresses and modify the mechanical properties of the film. This is precisely what is searched here : thermal annealing permits to densify the PECVD silicon nitride thin film, owning less internal stress than LPCVD one.

PECVD silicon nitride densification by thermal treatment takes its roots in two sources [54]. Firstly, PECVD SiN_xH_y films are known to be hydrogenous, hydrogen forming $Si-H_i$ (with $i = 1-3$) and $N-H_j$ (with $j = 1, 2$) bonds. The energy associated to those bonds being low, they can be broken by increasing temperatures. Bond breaking produces molecular hydrogen and induces the formation of $Si\text{--}Si$ and $Si\text{--}N$ in the network. It appears that the dissociation temperature of the $Si-H$ bond is around 350 °C, and around 1000 °C for $N-H$ bonds. Deshydrogenation is so the root of densification process, increasing the density of $Si-Si$ and $Si-N$ bonds. In plane internal stress emerges thus from the volume reduction associated to densification.

4.1.6 Thermal oxidation

Thermal oxidation is used to grow an oxide layer by oxidizing the surface of the substrate. In opposition to PECVD oxide deposition, a part of the substrate is transformed in oxide, by diffusion of an oxidizing agent into the wafer, followed by a reaction between this agent and the substrate. Thermal oxidation can be either dry or wet, depending of the presence of hydrogen or not. Almost all thermal oxidations preformed in the present process are dry ones, occurring at 900°C, following the reaction :



4.1.7 E-beam evaporation

E-beam evaporation, or electron beam physical vapor deposition, is a simple, fast and cheap microfabrication technique used to deposit a high purity metallic thin film on a substrate. This deposition is performed by bombarding the target material, lying in a crucible, with an electron beam. These electrons are produced by a charged filament and guided by an electro-magnet. The energy brought by these electrons serves to evaporate the material. Once the vapor phase is reached, the atoms or molecules precipitate on the substrate, and so form a coating layer on the wafer. This process is performed in a vacuum chamber (pressure about 10^{-7} mbarr) to give metallic atoms a ballistic trajectory by increasing their mean free path. This deposition method causes less damage than sputtering.

E-beam evaporation is used here to deposit nickel actuators (and chromium as adhesion layer). The non-conformality of this techniques makes it appealing for lift-off steps, by taking benefits from shadowing effects occurring during deposition.

The first step for a metallisation is to reach a chamber pressure around 10^{-7} mbar. Once done, one has to establish the deposition parameters, linked to different phases. To understand their meaning, one has to present briefly the deposition process. The E-gun power will be determined by the desired deposition rate. To reach it, the power has thus to go through a transient phase during which a shutter impedes gaseous metal to reach

the wafer. The e-gun power firstly increases by following a ramp until to reach a plateau to stabilize the power. The power increases then following a second ramp, during which the metal evaporates (or sublimates in the case of chromium), before reaching a second plateau, at a power level called the *predeposit power*. This parameters serves as starting point by the system to reach the steady power associated to the desired deposition rate. It has to be fixed manually and is mostly determined by previous depositions with the same material. Based on this value, the power will fluctuates until the deposition rate is stable. The shutter opens and the gaseous metal is deposited on the wafer. **Figure 4.5** represent those steps in a graphical way. The deposition rate is measured by piezoelectric sensing using a quartz crystal : when atoms reach the crystal surface they deform it and modify ts resonance frequency. This changes permits to extract the deposition rate on the crystal. The ratio between the quantity of matter deposited on the crystal and on the wafer is called the *tooling factor* and has to be manually calibrate. In addition to the predeposit power, the tooling factor and the deposition rate, the thickness of the layer has to be specified.

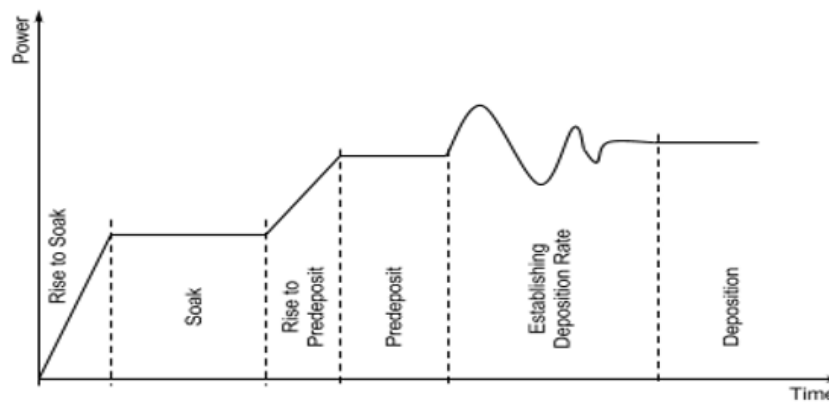


Figure 4.5: Graphical representation of the power as a function of time during the transient phase to reach the desired deposition rate. Different phases are mentioned. Graph provided by WINFAB team.

4.1.8 Critical point drying (CPD)

Critical point drying is a technique performed just after the release to avoid the stiction of the free standing thin film on the substrate. Indeed, during the liquid-vapor phase transition, capillarity force induces an adhesion of the two components. To avoid this problem, one uses the supercritical state of CO_2 . Fluids in a such state cannot be distinguished from neither liquid and gas. During CPD, the IPA in which the structure is immersed is replaced by liquid CO_2 , and the pressure and the temperature of the system are successively changed to turn around the critical point in the phase diagram of CO_2 (**Figure 4.6**).

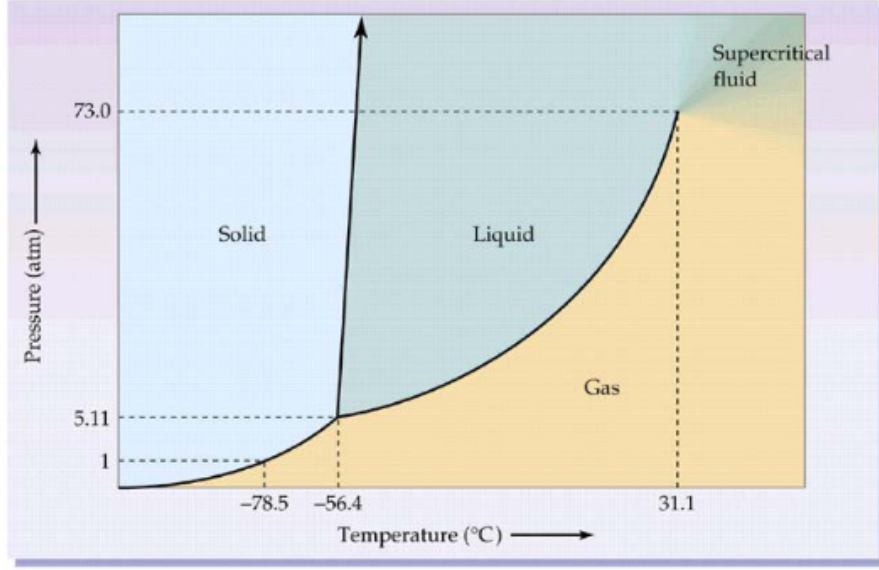


Figure 4.6: Phase diagram of CO₂ [55]

4.1.9 Characterisation techniques used along fabrication

Ellipsometry

Ellipsometry is a non destructive optical technique to measure the thickness of a single thin film or a stacking of thin films. Estimation of dielectric properties can also be obtained with this method, but its use in the framework of the master thesis is limited to thickness measurement. This technique is based on the change of polarisation of a linearly polarised light beam upon reflection by the surface of the sample. This change is evaluated through two parameters : the amplitude ratio Ψ and the phase difference Δ . The modification of these parameters upon reflection depends on the dielectric properties and thickness of the layers near the surface. The dielectric properties of deposited materials being available in database, the thickness of thin-film can be deduced by fitting the optical properties change with a model. Practically, the quantity measured by the ellipsometer and compared with a model is the complex reflectance ratio ρ , being a function of the two parameters introduced previously :

$$\rho = \tan(\Psi) e^{i\Delta} \quad (4.1)$$

By fitting the experimental curve and the model one, the thickness of each layer is deduced.

Profilometry

Profilometry is the second characterisation technique used during the present fabrication process, and consists in evaluating the surface profile of a sample. Practically, a diamond tip is put in contact with the sample and sweeps a defined distance by applying a constant force. Thanks to a feedback loop, the height is adjusted and a topography of

the sample along the probing trajectory is deduced. This technique is used for two purposes here : firstly, to evaluate the height of a step induced during a patterning phase, to deduce for instance a local etching depth. Secondly, to deduce the internal stress of a layer.

The extraction of internal stress present in a thin film is performed by evaluating the change of the wafer curvature due to the deposition of the film. Indeed, the temperature of thin films deposition is often higher than room temperature. During the cooling, the difference in the thermal expansion coefficient between film and substrate, the substrate has to curve to accomodate the strain. By making the hypothesis that the film thickness is negligible compared to the substrate one, the evaluation of the wafer curvature before and after the film deposition permits to estimate the internal stress of the deposited layer thanks to *Stoney* formula :

$$\sigma = \frac{1}{6} \frac{E_s}{1 - \nu_s} \frac{t_s^2}{t_f} \left(\frac{1}{R} - \frac{1}{R_0} \right) \quad (4.2)$$

Where E_s and ν_s are the Young modulus and Poisson ratio of the substrate, t_s and t_f the substrate and strained layer thicknesses, $\frac{1}{R}$ and $\frac{1}{R_0}$ the final and initial curvature of the substrate. Internal stress can thus be estimated without any information about thin film properties. The dependence of the substrate thickness in Stoney formula requires to perform a palmer measurement to get this information before processing the wafer.

Practically, the tip sweeps the entire diameter of the wafer and the maximal deflection of the tip h_{max} is deduced. The curvature radius can be expressed as :

$$R = \frac{L^2/4}{2h_{max}} \quad (4.3)$$

With L the distance probed[22].

4.2 Fabrication process of the labs on-chip

This section summarizes the fabrication process of the labs on-chip devoted to biaxial loading of silicon thin film. The role of each important step is described and the operation parameters are selected accordingly. Notable incidents occurring during the process are mentioned when needed. Some steps required some optimization of operating conditions to achieve the intended purpose. The main ones are available in the last section of this chapter, only results of those investigations are presented here when needed.

Unfortunately, strained structures were not obtained here due to some unexpected event at the end of the process. That is why the main results obtained in this fabrication sections mainly concern the informations obtained through these optimisation steps, added to a short evaluation of internal stress of investigated actuators by SEM measurements.

A schematic representation of the whole process is presented in the summary part after this section (**Figure 4.14** and **Figure 4.15**). The fabrication process can be divided in **4 key steps**.

- Wafer selection
- Specimen creation
- Actuators creation
- Release

4.2.1 Wafer selection

WINFAB facilities imply to use wafer of 3 inches diameter. Selected SOI wafers are made of pure monocrystalline silicon substrate, a silicon oxide layer constituting the BOX and a monocrystalline top silicon layer. The crystallographic orientation of the top-Si layer is pictured in [Figure 4.7 \(left\)](#). SOI wafers available in *WINFAB* and suited for the present investigation own a 1 μm -thick BOX layer and 140 nm-thick top-Si layer, n-doped with a charge carrier concentration of 10^{15} at/cm^3 . 700 nm-thick top-Si SOI wafer are also available, which provides an additional way to multiply the strain reached. However, due to time constraint, only the 140 nm top-Si ones were processed until the end, so the process is presented by considering those ones. The thicknesses of BOX and top-Si and native oxide layers are obtained by ellipsometry.

For the selected SI wafers (SUL5015 and SUL5016), the initial measurements give a 142.5 nm-thick top-Si/996 nm-thick BOX (for SUL5015), and a 144 nm-thick top-Si/997 nm-thick BOX (for SUL5015).

Throughout the process, standard monocrystalline silicon wafers are used firstly to check if the operation parameters of a particular step give the expected result, and secondly to evaluate the internal stress of a layer after depositions or annealing steps. In order to perform curvature measurements and to extract the internal stress, wafers with monotonic curvature has to be chosen. Indeed, taking a monotonic curved substrate is a first requirement to obtain accurate measurement. Monotonic and non-monotonic profiles are shown in [Figure 4.7 \(right\)](#). This measurements are performed by profilometry presented before. The use of a test wafer requires thus a preliminary profilometry measurement for the initial wafer curvature and palmer measurement for the wafer thickness.

4.2.2 Specimen creation

This step includes all the steps needed to go from the Top-Si layer to the cruciform specimens. The top-Si is patterned by reactive ion etching, The mask used for this etching is a photoresist one, presenting an acceptable selectivity compared to silicon when exposed to the plasma. After a **standard cleaning with HF**, the SOI wafers undergo a degassing step to ensure a good adhesion of photoresist during the spin-coating. In *WINFAB*, the **degassing** step is performed with the TEMPRESS oven set at 800 °C. The introduced wafer is placed between 4 wafers to ensure a uniform temperature profile. Ordinarily, the wafer is progressively introduced under a nitrogen flow of 2 liters/min, during 1 minute to avoid thermal shock. The flow is then decreased to 1.5 liters/min during 15 minutes.

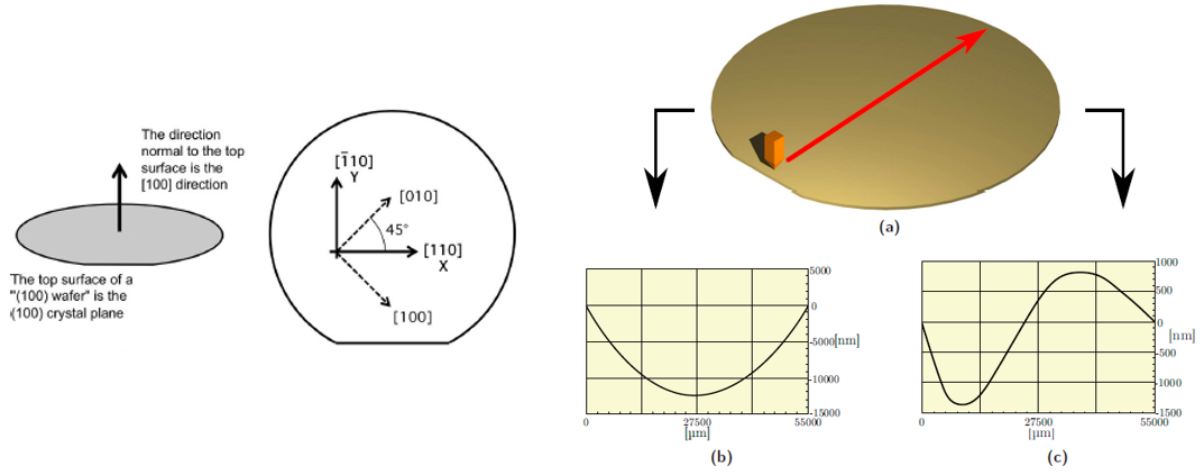


Figure 4.7: Wafer selection features. On left, cristallographic orientation of monocrystalline top-Si layer of SOI wafers [14]. On right, curvature measurement by profilometry of a standard wafer in the case of (left) monotonic and (right) non-monotonic profiles [22].

The nitrogen flow valve being damaged, degassing steps are performed with a constant 0.5 liter/min N_2 flow. This reduced flow can lead to the growth of a native oxide on the wafer surface. Once removed, the wafer is cooled down during a minimum of 3 minutes.

The photoresist is then spin-coated without delay on the wafer, with a velocity of 4000 rpm. The positive photoresist here is the AZ©MIR 701 SERIES, owning a sub-micronic resolution. A prebaking is performed at 90°C during 90 seconds. Photoresist thickness is about 880 nm. This prebaking serves to dry the photoresist and to stabilize it. **SPECIMEN** light field lithography mask is then put in vacuum contact with the wafer. This vacuum contact permits to limit the resolution decrease due to light diffraction, which may occurs in proximity contact⁴. This lithography step being the first one, no mask alignment is required. Exposure dose is set to 140 mJ/cm^2 . The exposure light wavelength, at which photochemical reactions occurs in photoresist, is 365 nm. A post exposure baking (*PEB*) is then performed (90 seconds at 110°C) in order to ensure a smoother and steep sidewall⁵. The development is performed with the AZ©300 MIF developer, during 60 seconds. Ellipsometry and optical microscopy are used to check the quality of the lithography. This step is critical because the photoresist mask is mainly responsible of the specimens shape accuracy. Except a poor quality of the lithography in lower instance, the other instances are well defined (see Figure 4.8).

A **reactive ion etching** is then performed to remove the silicon in the unmasked regions. The device used in for *Si* RIE is the *CORIAL 210 IL ICP-RIE*. Practically, the wafer to etch is placed on a plate surrounded by a ring, either in quartz or graphite. Due

⁴Wafers being common to biaxial tensile and shear testing, the shear structures present 0.75 μm details, compelling to use vacuum contact mode.

⁵Needed in the case of reflective substrate due to the interference occurring during monochromatic light exposure, leading potentially to some inhomogenity in concentration distribution of the photochemical reaction products

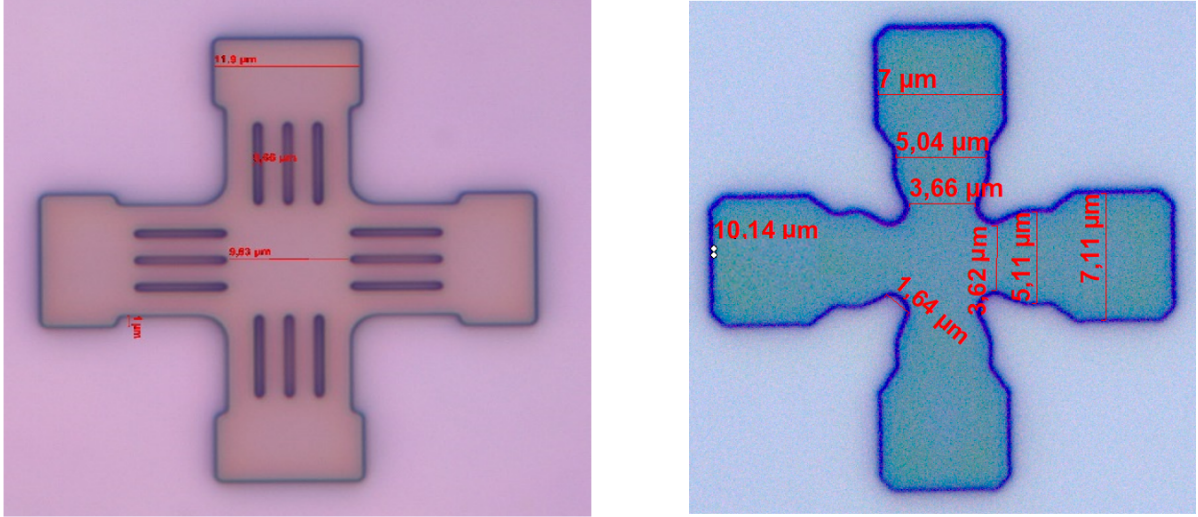


Figure 4.8: Optical microscope images of SOI wafers after photolithography dedicated to specimen creation.

to the organic nature of photoresist, graphite ring has been used in this work to reduce edge effects during the etching. All the etching steps performed with this machine follow the same procedure :

- **Chamber and ring cleaning** : the chamber is charged with a random standard wafer. During ordinarily 20 minutes, at 20 °C, a plasma composed of 15 sccm CHF_3 and 100 sccm O_2 is used as plasma cleaning. Due to the damage of the outer ring, the heat evacuation goes wrong so the time devoted to the cleaning step has been reduced in this work from 20 to 5 minutes to avoid considerable temperature increase. The cleaning pressure (10 mTorr) is a bit higher than the etching one to increase the number of collision with random direction, to give the cleaning a more isotropic character. In the same purpose, the ICP power is set to 800 Watt and the RF one to 0 Watt. This step also aims to put the chamber at temperature.
- **Chamber conditioning** : This step aims at tuning the capacities values to decrease the reflective power. The origin of this reflective power comes from the mismatch between the 50Ω-impedance feed line and the impedance of the chamber, due to the lack of auto-matching. A part of the power is so reflected and cannot be transmitted to the plasma, reducing the system efficiency. Practically, it is done by introducing a standard wafer in the chamber and performing an etching with the suited gas recipe. Capacitance values are tuned during this preliminary etching to set reflective power close to zero.
- **Etching** : besides gas flow (CHF_3/SF_6) composition, operating conditions are the same for all the etching in this master thesis : the temperature is set to 20 °C, RF power to 50 Watt and ICP to 800 Watt. The wafer is fixed to the back electrode by applying a clamping pressure, set at 4 mTorr. This pressure is maintained by varying the helium flow injected on the backside of the wafer (varying typically from

9 to 12 sccm). The etching time is then selected based on the etching rate⁶

In the present case, the sidewalls of the specimen have to be steep : this requires a highly anisotropic etching. SF_6 is here the etchant gas and CHF_3 the precursors provider for passivation (see the reminder about techniques). Recipe optimisation (see [Section 4.4.1](#)) leads to use a gas flow of 45 sccm CHF_3 / 12 sccm SF_6 for a highly anisotropic etching. Ellipsometry performed between successive wafers permits to adjust the etching time, allowing to complete the etching of the top-Si layer without etching too deep the BOX (otherwise the step between specimen and actuator is increased, reducing the efficiency of the overlap). The optimized etching time turns out to be 21 seconds for the present 140 nm-thick silicon specimens.

After performing check using optical microscope, remaining photoresist is removed by a **wet stripping**.

A way to enlarge the strain range present in the released specimen is to performed a **local thinning of the gauge**. The transition of stress level between gauge and out-of-gauge region should be as smooth as possible : it requests so a weakly anisotropic etching. Practically, three ways can be used : a *TMAH* wet etching, a CHF_3/SF_6 *RIE* or a XeF_2 *RIE*. Indeed, *TMAH* wet etching is isotropic and introduces tilted flanks in silicon perpendicular to the four $\langle 1\ 1\ 1 \rangle$ directions. This solution requires a SiO_2 mask, and some preliminary tests to deduce the *TMAH* concentration leading to a convenient etching rate. It has been noticed that a too high etching rate induces some strong roughness, not convenient for the present case [56]. Moreover, the etching profile obtained being pyramidal, stress will concentrate on the created edges, decreasing the distribution homogeneity. XeF_2 has the ability to etch isotropically silicon. However, this etching induces also a certain surface roughness. This leads to the third possibility : a CHF_3/SF_6 *RIE* with operating condition inducing tilted flanks. This is achieved by setting these gas flows at 50 sccm and 5 sccm respectively. The CHF_3/SF_6 is increased because a higher passivation is needed to obtain tilted flanks (this choice is also justified in [Section 4.4.1](#)).

This gauge thinning step begins by a standard cleaning (without *HF*), followed by a 800°C degassing and a photolithography with usual steps. **THINNING** photolithography mask being a dark field one, the exposure dose is slightly increased and set to 160 mJ/cm^2 . Lithography step is a critical step due to the required mask alignment : thinned regions have to be at the center of the structure. After confirming the quality of photolithography and checking the right alignment between gauge and specimen with optical microscopy ([Figure 4.9, left](#)), the reactive ion etching is performed with the *Corial* device following common procedure. The etching time is set to 10 seconds in order to etch 30 nm of the top-Si⁷. After microscope checking ([Figure 4.9, center and right](#)), remaining photoresist is removed by a stripping step.

The uniformity of the silicon plasma etching steps with *Corial* device can be discussed.

⁶A short transient phase existing before the steady one, the exact etching time is never exactly known.

⁷This value was chosen quite arbitrarily, one could have chosen to perform a deeper etching (a 50 nm thick specimen is considered as lower limit).

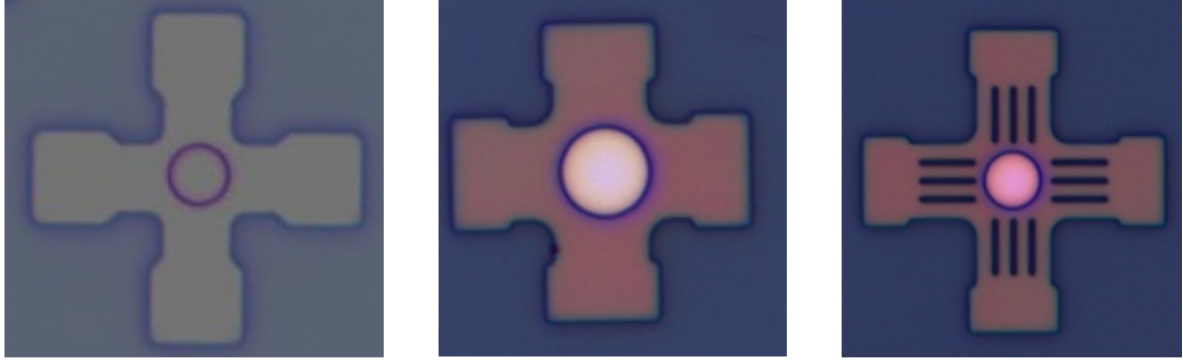


Figure 4.9: Optical microscope images of SOI wafers after (left) photolithography dedicated to gauge thinning. On center and right, images after the gauge thinning.

Etch rate appears to be larger near wafer extremities than near center. By performing a mapping after the gauge thinning step, we reported remaining top-Si thickness between 110 and 112 nm near the center of the wafer, and between 98 and 101 nm near the extremities for the SUL5015 SOI wafer. Same trend appears for the SUL5016 SOI wafer, where we measured 118 nm near the center and between 105 and 107 nm near extremities. This may be justified by the location of the gas flow entrances. A consequence is to obtain a different level of strain for structures located near the center of the wafer compared to those located near extremities for similar in-plane dimensions. The BOX was not impacted by the etching (996 nm-thick for SUL5015 and 997 nm-thick for SUL5016 after thinning).

4.2.3 Actuator creation

Usually, LPCVD Si_3N_4 is the material used as actuator for the labs on-chip developed at UCLouvain. Unfortunately, the *KOYO* device used to deposit this layer was out of order, and subcontracting this deposition by an external firm will induce some perturbation in the time schedule of the master thesis. Alternatives had to be investigated to tackle the unavailability of LPCVD Si_3N_4 , fulfilling the actuator specifications presented in the state of the art and being "WINFAB compatible". Three potential candidates were identified :

- **PECVD Si_3N_4** : this silicon nitride is less dense than the LPCVD Si_3N_4 . Its internal stress are expected to reach at maximum 500 MPa as-deposited and so to be lower than the LPCVD Si_3N_4 ones (about 1.1 GPa). A thermal annealing step can be done to increase them. Moreover, it has been noticed by *WINFAB* users that the thermal annealing also improve the PECVD Si_3N_4 resistance to *HF* (*WINFAB* users reported a 9 nm/min etch rate in *HF*-49% for LPCVD Si_3N_4 versus 0.5 nm/min for 2h at 950 °C annealed PECVD Si_3N_4). PECVD also permits to deposit slightly thicker layers than LPCVD.
- **Nickel** : metallic actuators are patterned by lift-off. Nickel is prone to creep, inducing a time dependency on the stress applied to the specimen. Its expected internal stress are greater than 1 GPa [25]. Nickel is known to have a non-negligible etch rate in *HF* (it was especially the case for releases performed with *HF*-73%). Nickel thicknesses previously deposited with *WINFAB* facilities never exceed 150

nm, and the thickness needed for the 140 nm top-Si SOI wafers has to be between 300 nm thick to ensure a good coverage between actuator and specimen extremities. This might thus be challenging. Moreover, in opposition to Si_3N_4 , an internal stress gradient is present across the nickel layer cross-section [57], leading potentially to out-of-plane deformation during release if nickel actuators are considered. Nickel being an electrical conductor, electrical measurements can be directly performed, with a suited annealing to create an ohmic contact between silicon and nickel, and with a suited photolithography mask, containing both actuators and electrical tracks pattern.

- **Platinum** : also patterned by lift-off. Platinum owns so high internal stress that auto-rupture occurs, even if deposited in ultra-thin thickness. However, it is not etched by HF . This choice is very expensive.

We have decided to investigate the PECVD Si_3N_4 and Nickel routes, the first is chosen with the "security" argument and the second one offers the opportunity to try a metallic actuator. The two following points are dedicated to each choice. For each route, a standard silicon wafer, called in the following *test wafer*, is selected and will undergo all the deposition steps with the SOI wafers. This allows to deduce the evolution of the internal stress present in the actuator material.

PECVD silicon nitride actuators route

The creation of PECVD Si_3N_4 actuators presents quite similar steps than LPCVD Si_3N_4 ones : firstly, a thermal oxide has to be grown on the specimen, playing the role of stopping layer for SiN etching. This stopping layer has to present some opening windows where actuator and specimen overlap. A photolithography step is thus required, followed by a wet etching to create these windows. Silicon nitride is then deposited, a photoresist mask is patterned and unmasked silicon nitride is etched by RIE.

The stopping layer is created by a thermal oxidation of the wafer in the *Koyo Thermo Systems* furnace. Indeed, a thermal oxide is preferred to a PECVD one due to its higher density, constituting a more resistant stopping layer. The oxide thickness has to be about 30 nm : the selectivity between SiN and SiO_2 is good in SiN plasma etching, and a larger thermal oxide thickness induces a larger thinning of silicon specimen. A preregistered recipe *DRY950 CMOS 27 nm* is used. From experience, this recipe leads to an oxide thickness of about 24 nm⁸.

The next step is to etch the thermal oxide at the opening windows. Wafers have the particularity to present good adhesion properties a few minutes after their extraction from the *KOYO* furnace, they are thus directly spin-coated with the positive photoresist without degassing step. Then follow the classic lithography steps : prebaking, exposure with the dark field mask **OVERLAP** (an exposure dose of 150 mJ/cm^2 is selected, the resist

⁸No ellipsometer check has been performed on test wafers after oxidation to extract the exact oxide thickness, but this oxidation step being well harnessed, the thickness should be really close to 24 nm. Moreover, the fit during an ellipsometry measurement with one of these test wafer by setting the oxide thickness to 24 nm was excellent.

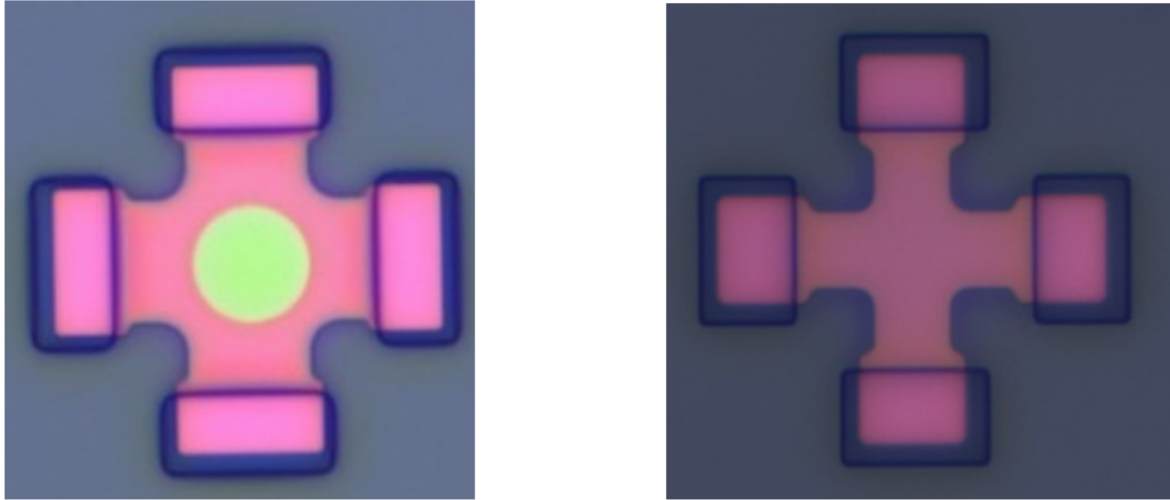


Figure 4.10: Optical microscope images of SOI wafers after photolithography dedicated to window opening for specimen/actuator overlap.

has not to be overexposed, because if the separation between two successive overlapping region is retrieved, the stress transmission will inevitably go wrong, this justifies the change from 160, the standard dose for the dark field masks, to 150 mJ/cm^2), PEB and development with common operating conditions. This step is also critical, due to the need of an accurate alignment of the mask to place overlapping regions at expected positions. Optical microscope images in [Figure 4.10](#) confirm the good quality of the lithography and the good alignment.

The silicon oxide present on the unmasked overlapping regions is then etched by *BHF*. This etching begins by a wetting, followed by a 30 seconds dip in *BHF*, which is known to etch 70 nm of SiO_2 per minute. The wafer is then rinsed by 5 fill/empty cycles in DI water, followed by a 5 minutes immersion in DI water with N_2 bubbles. An ellipsometry measurement is then performed to check that no oxide remains in the overlapping regions.

The stopping layer being created and windows opened, the PECVD silicon nitride can be deposited. This step begins by a standard cleaning and a short dip in *HF* to etch the native oxide present in the overlapping regions. The SOI wafers are then charged in the *Oxford Plasmalab 100* for deposition. A classic deposition is performed in three steps :

- A 10 minutes **stabilisation** step to put the chamber at temperature
- A 5 minutes **passivation** step with a well known deposition recipe to condition the chamber and to check if the plasma is well created.
- The **deposition** step : the PECVD *SiN* thin-film is created at the surface of the wafer, using SiH_4 , NH_3 and N_2 as incoming gas flow in particular proportions.

The passivation recipe here is the same than the deposition one. Gas flows are 1000 sccm SiH_4 , 21 sccm NH_3 and 1500 sccm N_2 , a power of 30 Watts and a temperature of 340

°C⁹.

Optimizing the deposition operating conditions was required to get high internal stress PECVD *SiN* film after annealing (see [Section 4.4.2](#)). Unfortunately, the *Oxford* device broke down just before the intended deposition. The problem appears to be caused by a massive clogging of the deposition chamber. The dirt level in the chamber was so high that the massflow was modified, leading to a silicon nitride with a different composition for the same input parameters before and after this cleaning. The major problem is that all the optimisation steps were performed for the *PECVD* *SiN* obtained before this manual cleaning. Performing a new optimisation of recipe/annealing condition being time consuming, it was decided to keep the same gas flows than before the change. Optimisation mentioned in the following refers thus to the silicon nitride obtained before the cleaning.

This optimisation led to a convenient recipe using 1000 sccm *SiH₄*, 21 sccm *NH₃* and 1500 sccm *N₂* as gas flows, a power of 30 Watts and a temperature of 340 °C. Indeed, with a 15-minutes thermal annealing at 600 °C, investigations led to internal stress about 1060 MPa. The desired thickness of silicon nitride layer is above 400 nm, slightly more than the one reachable by LPCVD, so the deposition time is set to 20 minutes. Ellipsometry permits to measure the silicon nitride film thickness on the test wafer SiN07 : 453 nm. A Stoney measurement is performed on this test wafer, which has also undergone the same oxidation and PECVD *Si₃N₄* deposition than SOI5016. Tensile internal stress are estimated at 172 MPa.

To increase the internal stress, an annealing step is performed to densify the silicon nitride. This step being followed by a photolithography to give *SiN* the actuators shape, A standard cleaning without *HF* is firstly performed. Preliminary tests makes us decide to perform this annealing at 600°C during 15 minutes (see [Section 4.4.2](#)). Indeed, highest internal stress were obtained by using this annealing temperature for test wafers, without the apparition of any fracture. A second Stoney measurement is performed on the test wafer to determine the internal stress evolution. The measurement on the SiN07 wafer test curvature gives an internal stress of 946 MPa.

A disappointing observation is the apparition of some cracks in the silicon nitride layer. A pertinent explanation may be that neither the PECVD *SiN* recipe was optimised after the chamber cleaning, nor the thermal annealing. Some small structures seemed to be not impacted by the cracks, so it was decided to continue the process with this SOI wafer.

The next step is to create a photoresist mask for the *SiN* patterning. Classic photolithography requires a degassing step at 800°to ensure the photoresist adhesion. The low temperature annealing replaces this step by using an adhesion promotor, functionalizing the wafer surface leading a better adhesion between wafer surface of photoresist. This avoids the apparition of additional cracks in the wafer. A photolithography with classic parameters is done, the photomask used is the light field **ACTUATOR** one, and the

⁹A manual cleaning of the chamber has been performed just before the deposition. The procedure in this case is to begin with a preliminary passivation step of 20 minutes at 300°C with a PECVD *SiO₂* recipe using 100 sccm *SiH₄*, 700 sccm *NH₃* and 1000 sccm *N₂* as gas flows

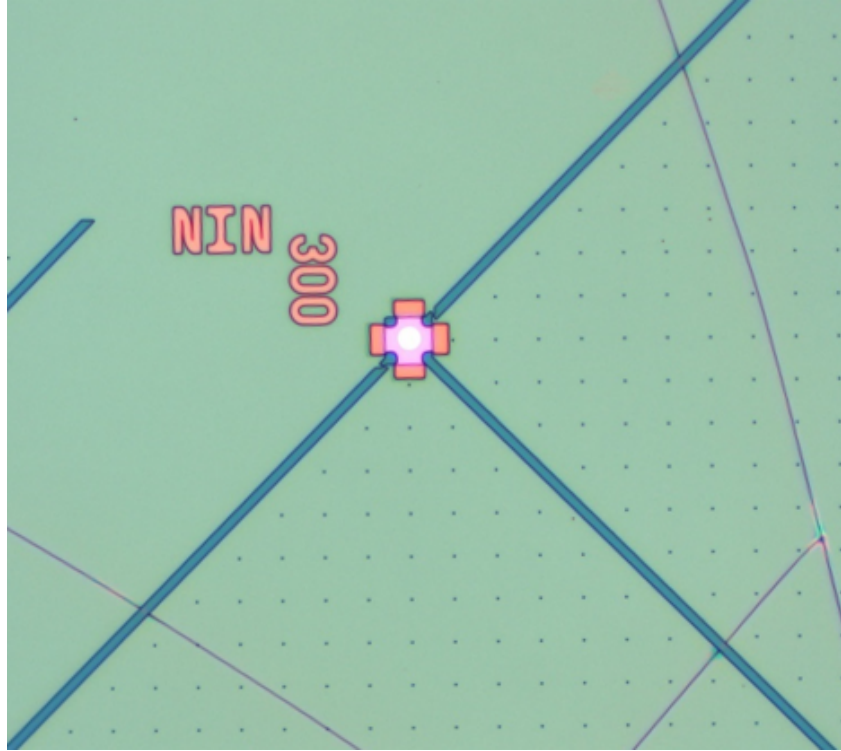


Figure 4.11: Optical microscope images of SOI wafers after SiN etching.

exposure dose is set at 160 mJ/cm^2 .

Actuators are then patterned by using the *RIE* module of the *Oxford Plasmalab 100*. Being an ICP RIE device, its working principle is roughly the same than the *Corial* device. The steps are the same than the *Si* etching ones :

- **Chamber cleaning** : the chamber and the plate are cleaned by a 15 minutes cleaning step. The cleaning plasma is created from a 100 sccm SF_6 /50 sccm O_2 gas flow, the chamber pressure being fixed at 25 mTorr (a bit lower than the etching pressure to induce a more isotropic cleaning due to the increase of the collisions with random orientation).
- **Chamber conditioning** : the etching recipe which will be used on the SOI wafer is selected and applied on a standard wafer to conditioning the chamber. This is also a way to ensure the creation of the plasma. The plasma used in the present case is a CHF_3/O_2 plasma, with a 100 sccm/10 sccm flow respectively. The ICP power is set to 0 Watt and the RF power to 100 Watt. This step is performed during 5 minutes.
- **Etching** : Recipe here is the same than chamber conditioning one. Investigations on a test wafer provides an etching rate of 11.3 nm/minute with the present conditions.

Optical microscope and ellipsometry are used to check that silicon nitride is completely etched at desired places. Figure 4.11 shows the SUL5016 SOI wafer after SiN plasma etching. Cracks are visible. The remaining photoresist is then removed by wet stripping.

Nickel actuators route

Nickel actuators are created by lift-off. The following process was tried on test wafers, following the procedure explained in [Section 4.4.3](#). After a standard cleaning, the SOI wafer is degassed at 800°C during 15 minutes. The lift-off negative photoresist being known to present difficulties concerning adhesion on the wafer surface, one proceeds to a surface functionalization. The spin coating angular velocity is fixed to 3000 rpm, the prebaking is performed at 90°C during 60 seconds, the exposure dose is set to $120\text{mJ}/\text{cm}^2$ ¹⁰ (light field **ACTUATOR** mask), the PEB of 60 seconds at 105 °C is then achieved, before a 60 second development.

The lift-off requiring a low conformal metal layer, the E-beam evaporation is a suited deposition method (if planar wafer holder is selected). The device dedicated to this task in *WINFAB* is the *E-gun Vacotec*. The planar wafer-holder is selected to decrease the conformality of the layer. The wafer are dipped in *HF* before being introduced in the chamber, to avoid the presence of any native oxide in the overlap region.

Nickel needing an adhesion layer, a chromium layer is firstly deposited. Classic adhesion layer being chromium and titanium, the chromium is the more resistant in *HF* (it is partially etched but pillars remains, ensuring the good adhesion). Chromium thickness is set to 3 nm and deposition rate to 1 Å/second. The tooling factor is set to 22. The crucible is cooled down, and the nickel evaporation is performed. A 300 nm thickness and a 2Å/second are selected¹¹. The deposition rate is an important parameter here because it governs the internal stress of the deposited metal by allowing or not an atomic rearrangement on the wafer surface. The crucible is then cooled, the pressure is progressively increased to reached the atmospheric pressure and wafers are extracted. Nickel being prone to creep, the Stoney measurement has to be performed without delay.

The wafer undergoes a lift-off in an acetone bath an ultrasonic vibrations. Some problems occurred during the lift-off step for the nickel actuator route. Nickel remained at some places normally coated with photoresist. We think that the same resist detachment occurred than the one presented in [Section 4.4.3](#). Nickel is so deposited as some unexpected places, leading to a poor lift-off with acetone. Hopefully, some instance was not impacted by this problem.

The test wafer (SiN06) dedicated to evaluate the internal stress present in the nickel underwent a thermal oxidation (thermal oxide thickness about 24 nm¹²). As expected, the presence of an oxide layer does not influence significantly the wafer curvature (the height of the valley is modified from -2499 nm to -2528 nm by the thermal oxidation). After metallisation, curvature measurements permits to evaluate the nickel internal stress at 580 MPa. It is slightly lower than the value obtained during preliminary tests (estimated at 625 MPa).

¹⁰Dose validate with preliminary tests

¹¹The speed are similar to the ones used in other micromachines fabrication

¹²the role of this wafer being not known at the beginning of the investigation, growing a thicker oxide layer, about 300 or 400 nm, should have been more relevant to imitate the BOX)

4.2.4 Release

The release step consists in etching the sacrificial layer to create free standing structures, allowing the stress transmission between actuators and specimen. The procedure is the following one : firstly, a particular instance of the wafer is extracted by cleavage and immersed in IPA. The instance is put in *HF* for the predetermined time, and is rinsed in three successive IPA baths (1 minutes with shaking in the first one, 5 minutes in the second and third ones). In the case of nickel actuators, one has to pay attention to avoid any contact with water to not induce the creation of some electrochemical couples. By keeping the instance immersed in IPA, the wafer is placed in the critical point drying holder, to perform a drying to avoid the stiction problem. The CPD is performed as explained in the reminder part.

The etch rate of actuator material being dependent of its process background, each release step begins by the determination of the present actuator etch rate and *SiO₂* etch rate. This permits to deduce the time needed to etch structures. Preliminary tests gave an *SiO₂* etch rate in *HF*-46% around 13 nm/second. Those tests were performed with optimised *SiN* layer before the manual chamber cleaning, but results are kept for the new silicon nitride. Nickel actuators turned out to be more resistant in *HF* as expected : after a 10 minute etch, its deterioration state was not considered as problematic (like it can be seen in [Figure 4.13](#)), so it was not judged useful to determine it (profilometry or cross section observation with SEM permits to get this information). Ordinarily, the etch rate is determined based on the dimensions of structures to release :

- for the narrow structures, the actuator large basis lengths is 20 μm . *HF* etching underlying *SiO₂* by the two etch fronts, those structures are released when both etch front has travelled 10 μm . With the present *SiO₂* etch rate, this corresponds to a 12 minutes 50 seconds etch. 102 nm of *SiN* are expected to be etched.
- for larger structures, etch time is governed by the etching hole spacing. 6.35 μm is needed to release the 5 μm x 5 μm specimens, and 12.7 for the 10 μm x 10 μm ones. This corresponds to a 8'8" and 16'16" etch time respectively. Theoretically, this leads to a *SiN* thickness decrease of 70 nm and 140 nm respectively. The silicon nitride thickness being around 450 nm, this decrease is quite important.

For the silicon nitride route, due to the cracks in actuator it was chosen to adapt the etching time to release only small structures. Etching time is so fixed to 10 minutes (we have just seen that 8'8" were theoretically sufficient but 10 minutes permits to bring a slight over-etch). A optical microscope check after the release led to a surprising and disappointing observation : the silicon nitride is completely etched ([Figure 4.12](#)). The change of the PECVD *SiN* recipe (and the impact on the thermal annealing) has certainly modify the etch rate in *HF*-49%, estimated at 8 nm/min for the recipe before the chamber cleaning. The thickness of the nitride layer being initially about 453 nm, the new etch rate is above 45 nm/minute now. Such a tremendous increase of etch rate was absolutely not expected. The etch rate of *SiO₂* appeared to be around 19.5 nm/second (11.8 μm of *SiO₂* etched), which is larger than the one determined during optimisation. Silicon oxide in BOX layer being similar to the thermal oxide growth in preliminary tests, it is unlikely that this discrepancy is explained by oxide variation.

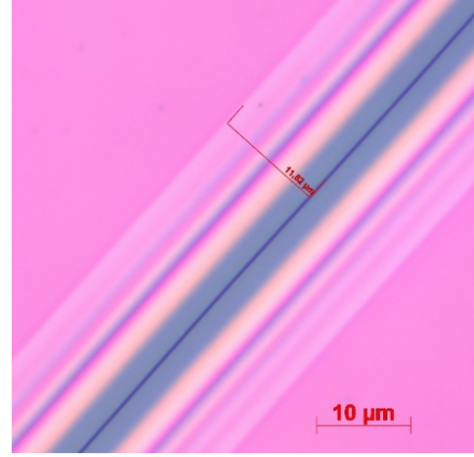
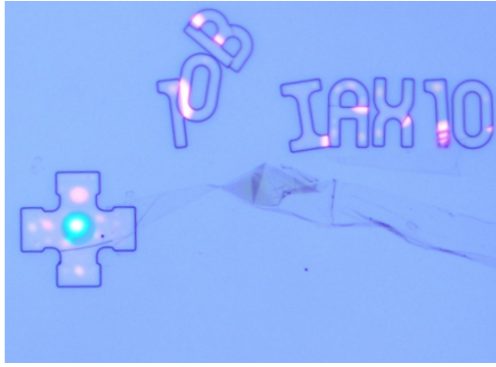


Figure 4.12: Optical microscope images of structures after final release. Right picture represents an under-etched silicon pad used to determined the etch rate of the present PECVD SiN

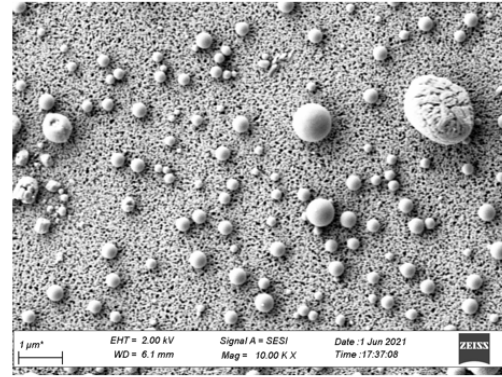
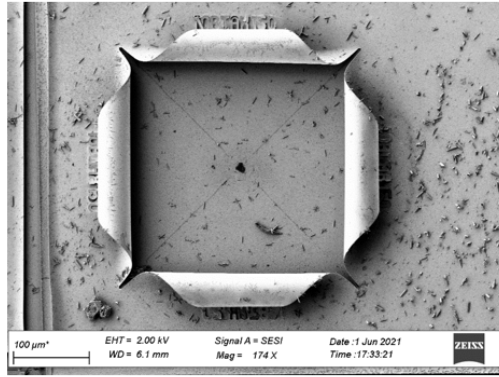
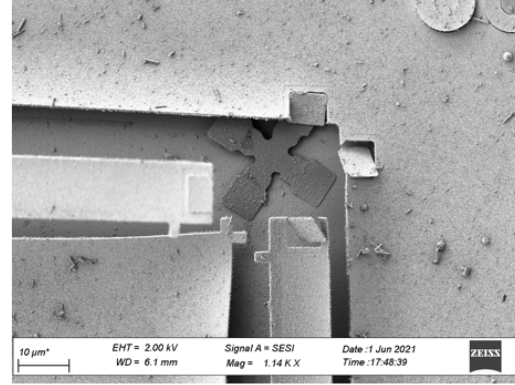
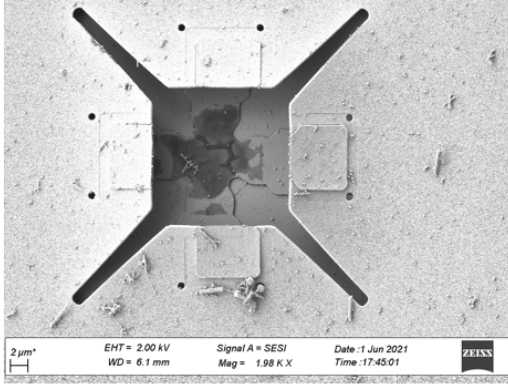


Figure 4.13: Structures with nickel actuator after release

For the nickel actuator route, a first instance was released 10 minutes in HF -49%. After performing a CPD, it was observed with SEM. Unexpected results were noticed, too. **Figure 4.13 (up)** shows the released structures. A detachment between specimen and actuators occurred, without inducing any damage to silicon specimen. A suggestion about what happen is that a native oxide had grown between the standard cleaning with HF

and the metallisation. Indeed, we do not clearly know the environment in which the wafer lies in the *VACOTEC* chamber before the pressure drop, a slight presence of oxygen can be sufficient to lead to a native oxide growth. This oxide is etched during the release, separating actuator and specimen. This explanation is strengthened by the silicon specimen on left, which seems to have turned once released and then dropped on the substrate after CPD. **Figure 4.13 (down)** shows on left the bending trend of nickel after release, and on right some impurities lying on the nickel surface, already obtained during preliminary investigations. SEM images allow to affirm that a 10 minutes release is enough to release the narrow actuator structures and the $5\text{ }\mu\text{m} \times 5\text{ }\mu\text{m}$ large actuator ones, as predicted theoretically.

4.3 Summary of fabrication

The process sheet dedicated to the fabrication of 140 nm-thick silicon specimen structures can be found in appendices ([page 124](#)). A schematic representation of the main steps of the two routes are schematized in **Figure 4.14** and **Figure 4.15**

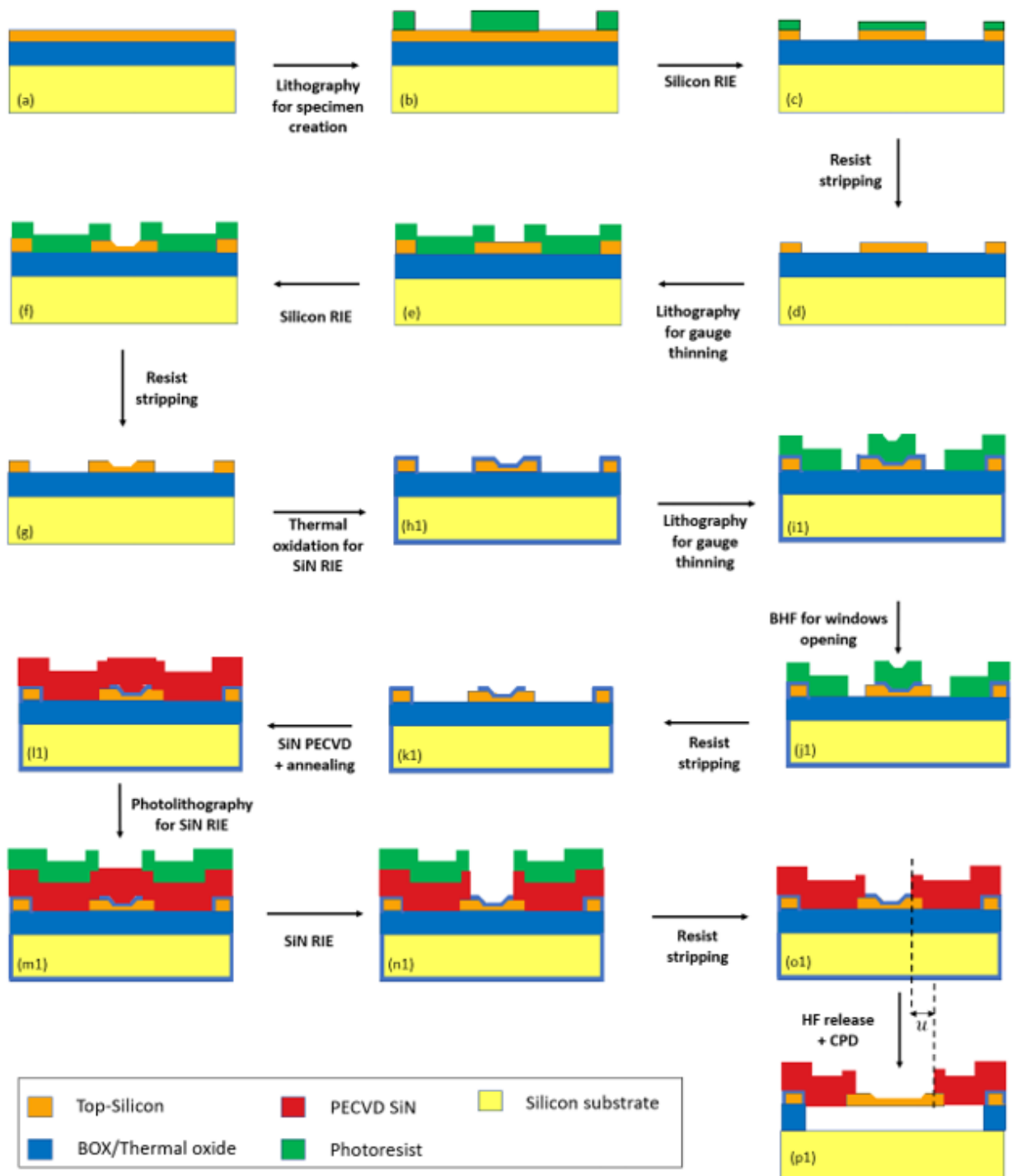


Figure 4.14: Representation of the process devoted to PECVD SiN actuator route.

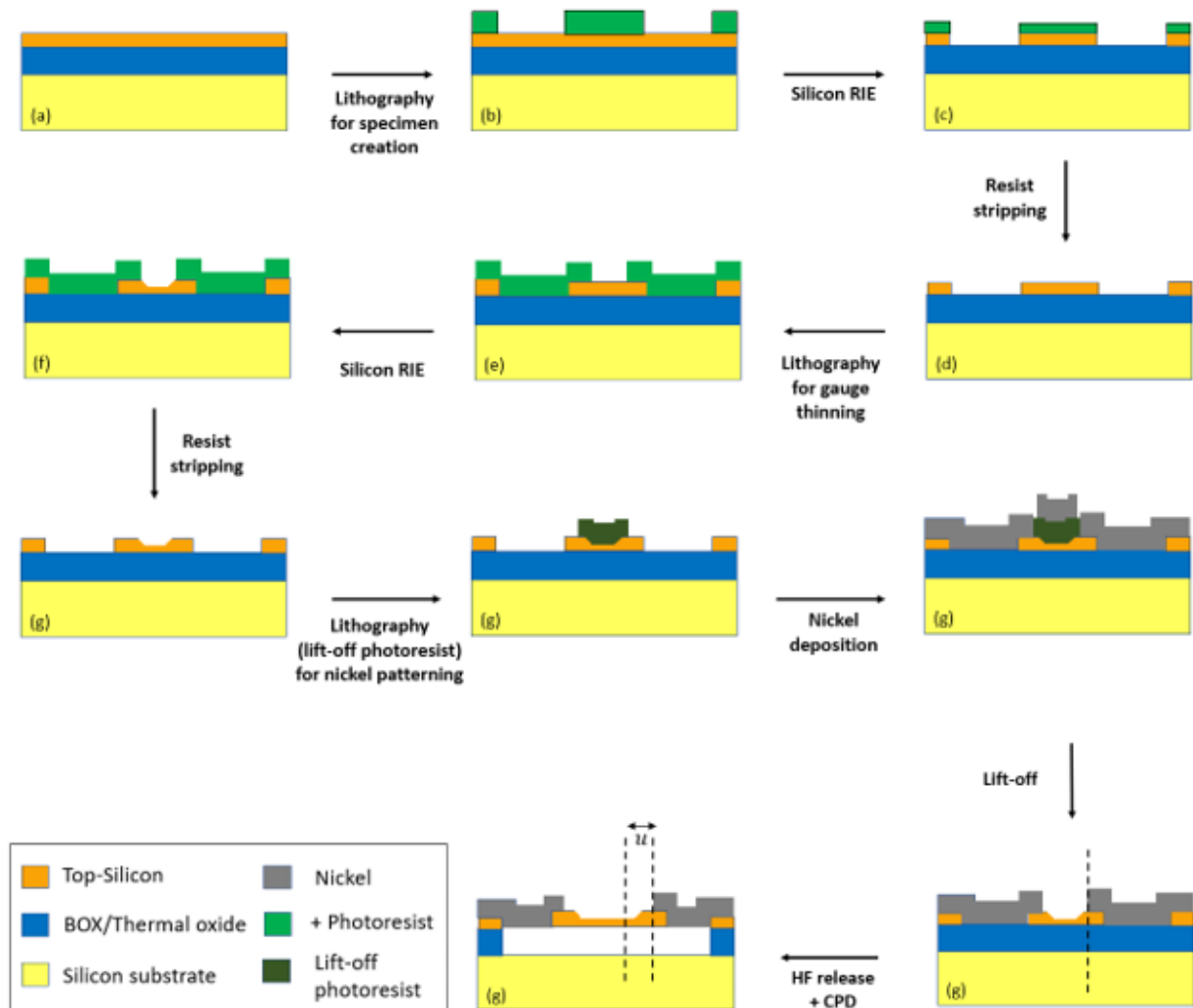


Figure 4.15: Representation of the process devoted to nickel actuator route.

4.4 Optimisation steps

Each step of the process aims at fulfilling a role. In this purpose, operating parameters have to be tuned and optimized to ensure to obtain the desired result when the step is applied to the real SOI wafer. Due to the cost and the limited availability of SOI wafer, standard 3 inches silicon wafers are used for those optimisations of operating parameters. In this section, investigation about RIE etching of silicon is summarized, in addition to the Si_3N_4 plasma enhanced chemical vapor deposition and thermal annealing, and actuator etch rate in HF . This last point required the fabrication of structures which will be also used to determine internal stress by SEM measurements, in the characterization chapter.

4.4.1 RIE etching of silicon

The *CORIAL 210 IL* device being quite recent in *WINFAB*, etching recipes for a desired purpose have to be derived by users. The present process needs both a recipe of silicon etching inducing steep (for specimen creation) and less steep sidewall (for gauge thinning). Only the gas flows are modified, powers are kept constant (both powers and gas flows impact etch rates and selectivity so keeping one variable constant simplifies to optimisation). For this investigations, standard silicon wafers coated with a photoresist layer (patterned with the light field *SPECIMEN* mask) are used.

- First RIE is performed with a **50 sccm CHF_3 /5 sccm SF_6** gas flow during 100 seconds. Ignoring at this stage the existence of a graphite ring, a quart ring WF75 was used to surround the wafer during etching. SEM images reveal a highly tilted flanks and a quite inhomogeneous etching. This inhomogeneity may be justified by the lack of continuity of carbonated species near the wafer interface (carbon/quartz transition), leading to edge effects. SF_6 being the etching specie and CHF_3 the passivation one, increasing the SF_6/CHF_3 ratio should give more vertical flanks.
- Second RIE is performed with a **50 sccm CHF_3 /10 sccm SF_6** gas flow during 75 seconds. Graphite ring is used. The situation is improved compared to the first try. **Figure 4.16 (left)** shows a cross section view of the etched test wafer. The anisotropy is around 0.2, which remains non negligible. The SF_6/CHF_3 ratio can be increased.
- Third RIE is performed with a **50 sccm CHF_3 /15 sccm SF_6** gas flow during 75 seconds. Flanks are quite more tilted than the precedent try, an a surprising trend is the presence of a high tilt difference between left and right flanks of structures. **Figure 4.16 (right)** shows a cross section view of the obtained tilted flanks.
- Until now, second investigation with a 50 sccm CHF_3 /10 sccm SF_6 gas flow gave the best results. Only the etching gas flow was increased. Let us see what happen if the passivation gas flow is slightly reduced while slightly increasing the etching one. The etching is so performed with a **45 sccm CHF_3 /12 sccm SF_6** gas flow during 75 seconds. Except some rare oddities, structures present well vertical flanks, really suited for the specimens. This recipe is so selected for specimens creation. **Figure 4.17** illustrates those vertical flanks. Si etch rate is around 10 nm/second and photoresist around 4.5 nm/second. The Si/PR selectivity of the species for the present plasma is thus around 2.2:1. For the profile of the specimen, knowing that

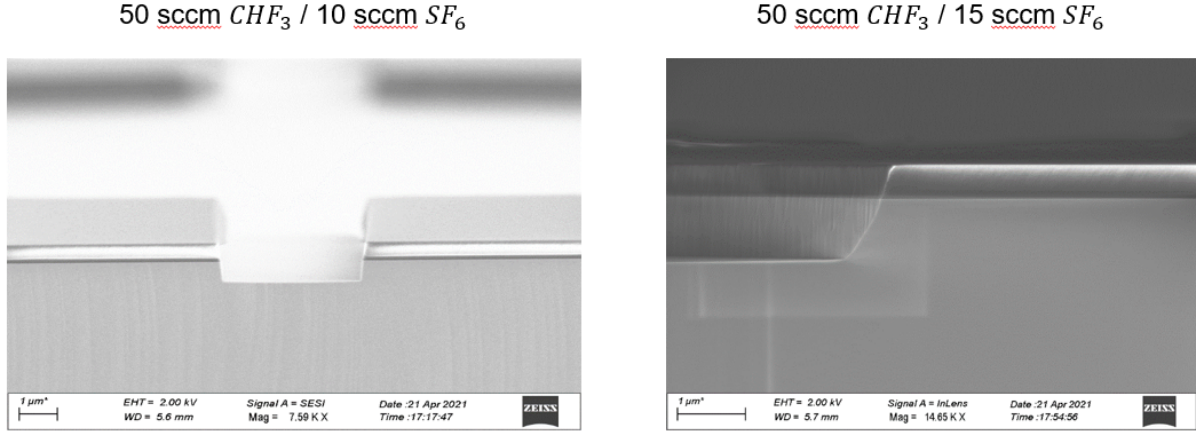


Figure 4.16: Cross section view on a test wafer etched by a 50 sccm CHF_3 /10 sccm SF_6 (left) and 50 sccm CHF_3 /15 sccm SF_6 (right) gas flow during 75 seconds.

the photoresist layer thickness is about 880 nm, this selectivity is large enough.

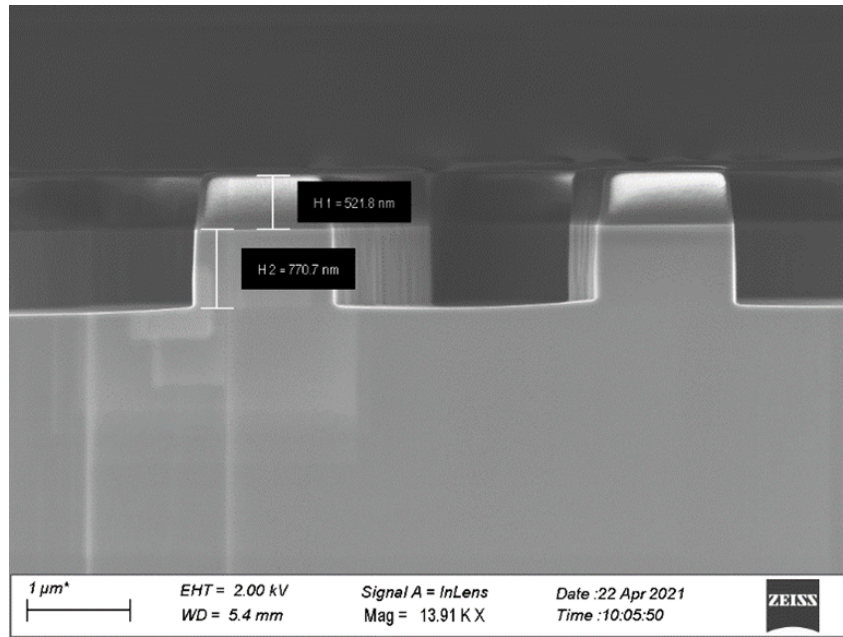


Figure 4.17: Cross section view on a test wafer etched by a 45 sccm CHF_3 /12 sccm SF_6 gas flow during 75 seconds.

Particular SOI wafer with 2000 nm-thick top-Si, used for structures devoted to shear load, requires multiple successive etch to create the specimen. This is a strong way to validate the developed recipe. However, one prefers to use a one shot etch to extract the etch rates and selectivity. The test wafer used for the fourth investigation is so etch a second time with the same recipe, during 140 seconds. Structures present similar vertical flanks with this additional etching, validating it for any silicon etching requiring high anisotropy (Figure 4.18 (right)). One notices however, for some structures, the presence of

a slope change, roughly situated at 2/3 of the silicon etch profile (Figure 4.18 (left)). This may be explained by the conditioning of the etching device through successive uses. The first third of etched region was performed during the first etch step (fourth try). Being the first etch achieved by the CORIAL device that day, only one conditioning step of the chamber was performed. The second etch step was performed after etching the whole set of SOI wafers (around 15 wafers) with the same recipe. The chamber having been conditioned in the same way several times, operating conditions were enhanced and this led to an even larger anisotropy. The etching depth here seem vary along the wafer axis, being larger near the extremity than the center. This trend is confirmed in the results parts with the SOI wafers.

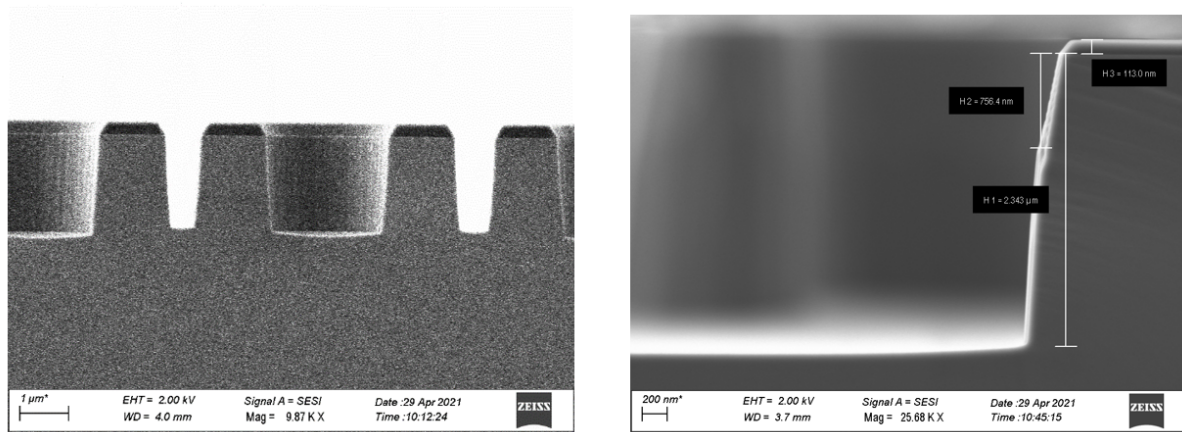


Figure 4.18: Cross section view on a test wafer etched by a 45 sccm CHF_3 /12 sccm SF_6 gas flow during 75+140 seconds. On left the particular case of a flank slope change. Etch depth are displayed.

For the gauge thinning, the first try recipe meets the specifications (50 sccm CHF_3 /5 sccm SF_6) and is so selected. Figure 4.19 shows the flank profile obtained after a 10 seconds etching.

4.4.2 Plasma enhanced chemical vapor deposition of Si_3N_4 thermal annealing

Due to the change of massflow in *Oxford* device, old recipes are not available anymore. One has thus to perform investigations to deduce a PECVD Si_3N_4 recipe and thermal annealing conditions leading to high internal stress in the SiN layer. A preliminary thermal oxidation is performed on all the test wafers (oxide thickness is around 24 nm). Results of investigations are shown in Table 4.1 .

Here is a quick recap of the methodology used. The first recipe tried (for wafer T04) is based on a recent recipe known to own internal stress. This recipe led to 150 MPa internal tensile stress for the as-deposited silicon nitride film. By performing a 35 minutes thermal annealing at 950 °C, internal stress increased until 970 MPa, which can be considered as suited. However, some cracks appeared in the silicon nitride layer, oriented along the

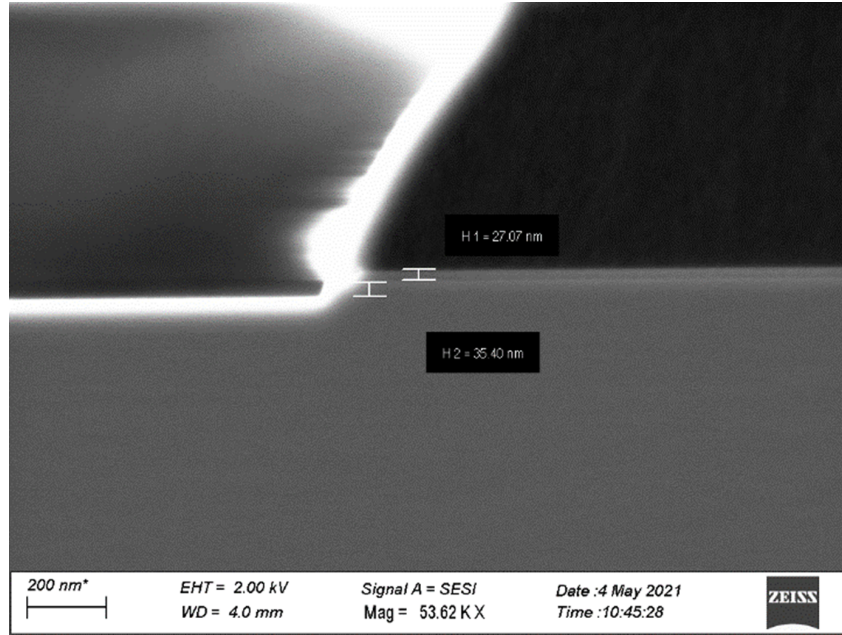


Figure 4.19: Flank profile of a test wafer etched by a 50 sccm CHF_3 /5 sccm SF_6 gas flow during 10 seconds

$\langle 100 \rangle$ direction, shown in Figure 4.20 (an attempt to justify this particular crack propagation direction is proposed further). To obtain high internal stress SiN without cracks, one has either to change the recipe or thermal annealing conditions. The same recipe is so used for a first wafer (R02), and knowing that internal stress is expected to increase by decreasing the SiH_4/N_2 ratio, the PECVD recipe is modified and applied to a second test wafer (R03). A 15 minutes thermal annealing at $800^\circ C$ is then performed, but cracks still appeared on the nitride layer. The etching step of SiN layer needing a photolithography step, decreasing the annealing temperature below $800^\circ C$ could be problematic for degassing step. Another PECVD SiN recipe is so tried, based on a successful one used before the massflow change, applied on test wafer T05. Even though presenting low internal stress after deposition (around 80 MPa), a $500^\circ C$ thermal annealing permits to reach 1040 MPa. To investigate the temperature at which cracks appear, successive annealing are performed, from 600 to $900^\circ C$, by step of $100^\circ C$. Stress measurements are performed between each step to estimate the internal stress change. Internal stress increases until $700^\circ C$ to reach a maximum value of 1060 MPa, before decreasing until $900^\circ C$ where cracks appeared, again along the $\langle 100 \rangle$ direction. To check if the precedent multiple annealing steps influence the internal stress level of the wafer, another test wafer (T07) is coated with the same SiN recipe and directly annealed at $800^\circ C$. The level of internal stress is lower than for the wafer undergoing successive progressive annealing, performing successive progressive annealing is so more efficient to reach higher stress levels. Wafers Si02 and Si03 validate stress level obtained with T07 with a 15 minutes thermal annealing at $800^\circ C$.

The particular operating conditions (especially precise gas flow ratios) makes comparisons to literature difficult because each research group works with a different silicon nitride. However, some similarities can be pointed out. Yang et al. investigates the

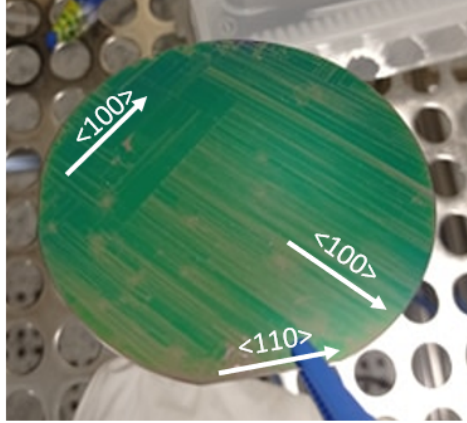


Figure 4.20: Cracks appearing in SiN layer after a $950^{\circ}C$ thermal treatment

evolution of PECVD SiN internal stress as a function of annealing time and temperature. They worked with a free internal stress nitride film, deposited at $200^{\circ}C$. Their results are summarized on the graph present in appendices (page 124). Firstly, the annealing duration of 15 minutes seem to be optimal when deposition temperature are larger than $500^{\circ}C$. After these 15 minutes, a relaxation occurs, decreasing the internal stress. The level of strained reached in their paper is in a general way lower than the one of the present investigation, which may be justified by the zero-stress state of as-deposited film. However, the stress evolution with temperature follows the same trend : a stress increase from $500^{\circ}C$ to $600^{\circ}C$, followed by a decrease until $700^{\circ}C$. The decrease of stress in our case may be also related to this relaxation process, Unfortunately, physical nature of relaxation process was not specified in the paper.

As explained before, performing a low temperature annealing can be problematic because the wafer undergoes a $800^{\circ}C$ degassing in a further lithography step. Two ways can be envisaged so : either choosing a annealing temperature of $800^{\circ}C$, or a lower temperature like $600^{\circ}C$ but using an adhesion promotor (LP3) instead of performing a degassing step before the lithography. The last route being more safe, it is chosen for the real SOI wafers.

Wafer name	Wafer thickness	Initial curvature	SiH_4	NH_3	N_2	T°	Power	Deposition time	SiN thickness	Curvature after dep.	σ_{int}^{dep}
	μm	nm	sccm	sccm	sccm	°C	Watt	min	nm	nm	MPa
T04	377	5935	600	21	700	300	30	14	326	1599	150
R03	390	-2291	300	21	700	300	30	20	416	-13646	331
R02	389	-2437	600	21	700	300	30	15	389	-8055	173
T05	382	7951	1000	21	1500	340	30	15	363	5377	80
T07	382	7951	1000	21	1500	340	30	20	474		
SiN02	389	2214	1000	21	1500	340	30	20	421	-3887	173
SiN03	390	1312	1000	21	1500	340	30	20	418	-5102	185

Wafer name	Wafer thickness	Initial curvature	SiN thickness	σ_{int}^{dep} after dep.	Annealing temperature	Annealing time	Curvature after annealing	$\sigma_{int}^{anneal.}$	Cracks ?
	μm	nm	nm	MPa	°C	min	nm	MPa	
T04	377	5935	326	150	950	35	-21393	970	Yes
R03	390	-2291	416	331	850	35	-50942	1400	Yes
R02	389	-2437	389	173	850	35	-30180	850	Yes
T05	382	7951	363	80	500	15	-24592	1040	/
					600	15	-25237	1060	/
					700	15	-20632	911	/
					800	15	-20482	908	/
					900	15	-19798	885	Yes
T07	382	7951	474		800	15	-26657	736	/
					900	15	-29737	811	Yes
SiN02	389	2214	421	173	800	15	-24888	772	/
SiN03	390	1312	418	185	800	15	-25878	784	/

Table 4.1: (Up) : Oxidized wafers used for *PECVD SiN* route investigation. *PECVD* operating parameters are mentioned and *SiN* internal stress is estimated. (Down) : evolution of *SiN* internal stress with different annealing temperatures.

Quite surprisingly, cracks along $\langle 1\ 0\ 0 \rangle$ directions appeared in the silicon nitride layer. PECVD SiN thin film being amorphous, the existence of preferential crack propagation directions is linked to the anisotropic nature of the underlying monocrystalline silicon. No papers reported this particular case in literature. However, a research group encountered the same problem with an analogous structure : a monocrystalline silicon substrate on which is electrochemically growth an amorphous silicon lithiate layer $a-Li_xS$ [58]. During the delithiation process (the amorphous layer is already completely grown), cracks appear along the $\langle 1\ 0\ 0 \rangle$ directions. The provided justification is based on the application of the LEFM theory on multi layer structures. Here is the application of this theory for the present case.

To apply LEFM relies, elastic fracture characteristics must appear at the crack location, like sharp edges and flat faces. This can be verified by SEM observations. According Linear elastic fracture model, a crack propagates in the film only if the energy release rate G , defined as the instantaneous loss of potential energy by unit of new crack surface created, surpasses the fracture toughness of the film Γ_C :

$$G = \frac{\pi(1 - \nu^2)\sigma^2 h}{E} g(\alpha, \beta) \geq \Gamma_C \quad (4.4)$$

Here, E is the Young modulus, ν the Poisson ratio, σ the plane stress, h the film thickness and $g(\alpha, \beta)$ a function depending on the elastic mismatch between the bulk and the top film. The Dunder's parameters α and β are defined with the plane strain elastic modulus and the shear modulus of the top film (-) and the substrate (s) :

$$\alpha = \frac{\bar{E} - \bar{E}_s}{\bar{E} + \bar{E}_s} \quad \text{and} \quad \beta = \frac{\mu(1 - 2\nu) - \mu_s(1 - 2\nu)}{2(\mu(1 - \nu) + \mu_s(1 - \nu))} \quad \text{with} \quad \bar{E} = \frac{E}{(1 - \nu^2)} \quad \text{and} \quad \mu = \frac{E}{2(1 + \nu)} \quad (4.5)$$

Equation (4.4) allows us to derive a critical fracture thickness corresponding to the thickness for which the release energy rate G and the fracture toughness are equal :

$$h_c = \frac{2 \bar{E} \Gamma_C}{\pi \sigma^2 g(\alpha, \beta)} \quad (4.6)$$

Given that σ and $\bar{E}$ are constant in this equation, the critical thickness is a function of $g(\alpha, \beta)$, known to increase with α (the dependence with β is weak). It can thus be concluded that the critical cracking thickness is governed by the plane strain elastic modulus of substrate and top film.

Silicon being anisotropic, its plane strain elastic modulus depends on the crystallographic orientation : by taking the Hopcroft values [14], the plane strain elastic modulus value in $\langle 1\ 0\ 0 \rangle$ direction is the lowest one ($\bar{E}_s = 148.38$ GPa) and in $\langle 1\ 1\ 0 \rangle$ direction the biggest one ($\bar{E}_s = 183.39$ GPa). By injecting these value in Equation (4.5) and with the similar evolution of $g(\alpha, \beta)$ and α , it comes that the largest value of the function $g(\alpha, \beta)$ is associated to the $\langle 1\ 0\ 0 \rangle$ direction, leading to the smallest critical thickness following Equation (4.4). Cracks are thus propagating preferentially along $\langle 1\ 0\ 0 \rangle$ direction, confirming the trend observed experimentally.

4.4.3 Determination of actuator resistance in 49%-HF + Fabrication process of structures devoted to internal stress measurement by SEM

In order to choose a convenient release time, actuator etch rate has to be estimated. This material parameter being quite dependent of the actuator fabrication process, one cannot limit to use standard values. For this purpose, standard wafers are selected : SiN05 for the silicon nitride route and Ni01 for the Nickel one. Structures devoted to investigate *HF* resistance and internal stress by SEM measurements requiring the same fabrication process, the present structures will be used for both purposes.

For the silicon nitride investigation, we grew a 24 nm thermal oxide on *SiN05* (playing the BOX role), before creating the actuator in a similar way that the one used for real SOI wafer. A 418 nm-thick PECVD *SiN* layer is then deposited with the optimised recipe, followed by a 800°C thermal annealing during 15 minutes¹³. A photoresist mask is then created on the surface, before performing a RIE of *SiN*. The etching recipe is the same than the SOI process one, and the etching time is set to 35 minutes¹⁴. Structures can now be released : lower instance being the worst one, this instance is dipped in IPA before being immersed in HF-49% during 230 seconds. Then we perform rinsing in a first IPA bath during 1 minute with agitation, then in a second IPA bath during 5 minutes. The instance is then rinsed and dried. Figure 4.21 shows optical microscope images of actuators after HF etching. The presence of pads where silicon is visible permits to deduce the thickness of *SiN* etch by measuring the difference of step between silicon and silicon nitride before and after release. Measurements leads to a decrease of 30 nm, leading to an etch rate of 8 nm/min for the present PECVD *SiN* in *HF* – 49%. To determine precise *SiO₂* etch rate and PECVD *SiN* internal stress, a second release step is performed with an higher quality instance, with the same procedure but changing the etch time to 260 seconds. Once released, this instance is kept in a watch glass filled with IPA, before undergoing a critical point drying. Figure 4.22 obtained by SEM observation permits to estimate that between 3 and 3.5 μm of thermal oxide are etched during this release, leading to a *SiO₂* etch rate between 11.5 to 13.5 nm/second. Rough measurements with optical microscope gives similar etch rate.

For the nickel route, a thick thermal oxide layer (about 394 nm-thick) is firstly grown on the Ni01 wafer surface in KOYO furnace, to imitate the BOX. This set having also the aim to determine the evolution of internal stress by Stoney measurements, another test wafer, SiN08, followed the same deposition steps. An LP3 is then applied to Ni01 before coating it with negative photoresist (at 3000 rpm), to perform a lift-off deposition. A 60 seconds/90°C prebaking is performed. An old WINFAB photolithography masks containing many auto-actuated structure is used for the exposure step, performed with a dose of 120 mJ/cm^2 . This is followed by a PEB (60 seconds/105°C) and a 60 seconds development. Nickel is deposited on Ni01 and SiN08 in a low conformal way by thermal

¹³At this stage, this annealing temperature was considered as the best one. This turned out to be not the case

¹⁴This step was the test of the etching recipe for SOI wafers. This etching time induces a slight over-etch.

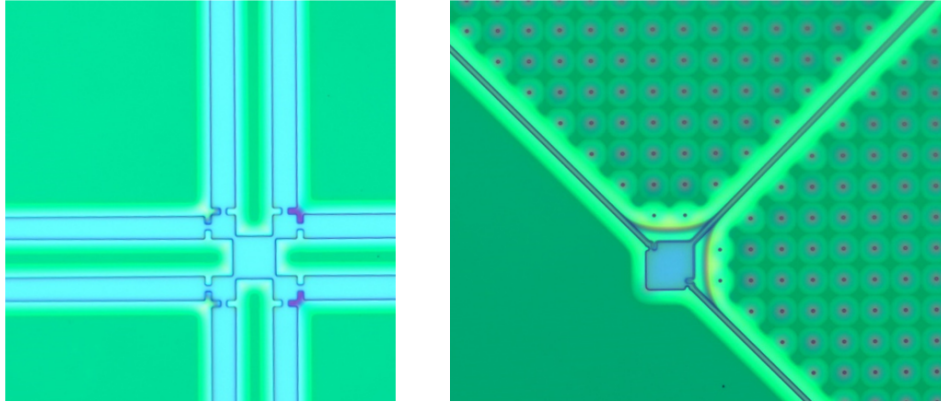


Figure 4.21: Optical microscope images of (left) narrow actuators structure and (right) large actuator structures for $5\ \mu\text{m} \times 5\ \mu\text{m}$ specimens, after 230 secondes in HF-49%

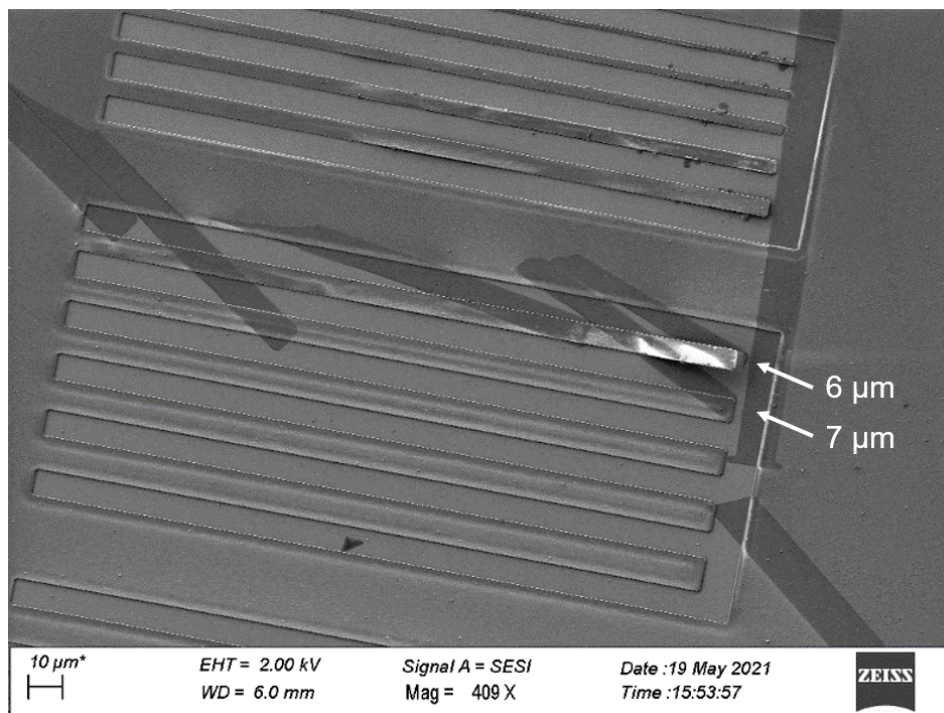


Figure 4.22: SEM image of quality structures, showing that between 3 and $3.5\ \mu\text{m}$ of thermal oxide are etched after 260 secondes in HF-49%

evaporation, with exactly the same procedure and operating parameters than the one used for the SOI wafer. A surprising result is observed after the deposition, as shown in [Figure 4.23](#). Here is an interpretation : during the metallisation, the internal stress of the nickel grew so much that it induces an out-of-plane bending of the nickel at some places. Nickel adhering well to the photoresist thanks to the adhesion layer, the underlying photoresist remains attached to the nickel and peeled off from the oxide layer. The lift-off is then performed in acetone, but went wrong at some places : nickel remains where it should not. This may be due to the fact that the photoresist detachment occurs whereas metallisation is not finished : nickel is so deposited on places not masked by photoresist

anymore, and cannot be removed during the lift-off. However, some instances looked good and are used for the release. Procedure is exactly the same than the one used for silicon nitride route, the release time is set to 260 seconds. Optical microscope check reveals that the damage of nickel due to HF is acceptable, no precise measurement of etch rate is performed. After a CPD, this instance is analysed using SEM.



Figure 4.23: Nickel layer after metallisation.

4.4.4 Optimization through SILVACO simulations : Doping the silicon

A motivation associated to the development of the present lab on-chip is to permit an electrical characterisation of biaxially strained silicon. This can lead to the extraction of materials parameters such as piezoresistivity for instance. Silicon being a semiconductor, limiting to intrinsic charge carriers requires to apply high voltages for property characterisation. A way to ease the characterisation is to increase the charge carrier mobility of the silicon by doping. Practically, such steps are performed in *WINFAB* by impurity diffusion through the surface of the wafer. Top-Si layer of SOI wafers are n-doped with a doping concentration of about 10^{15} at/cm³. In *WINFAB*, ion diffusion for n-doping can be achieved by evaporating phosphorous atom from a solid source in a particular furnace.

A common practice used for CMOS process in *WINFAB* is to perform two successive impurity diffusion of 30 minutes or one hour at 900 °C, separated by a standard cleaning to remove the phosphorous glass created on the surface. Other shorter phosphorous diffusion (15 minutes or 30 minutes) at the same temperature were also performed.

To derive convenient diffusion parameters (diffusion duration and temperature), *ATHENA* module of *SILVACO* software can be used. From literature, the top-surface impurity concentration is determined by the solid solubility of the layer to the involved impurity., which is, for phosphorous in silicon, around $2.9 \cdot 10^{20}$ at/cm³ [59]. To ensure a good electrical conductivity, one arbitrarily aims at reaching at least a charge carrier concentration of 10^{19} at/cm³ over the 100 first nanometers of the top silicon.

The system representing the SOI wafer is firstly built in *SILVACO*, the initial n-doping of silicon is set to 10^{15} at/cm³. The phosphorous concentration at near the wafer surface is set to $2.9 \cdot 10^{20}$ atom/cm³, corresponding to the equilibrium concentration at saturation as explained before. Doping center diffusion is represented by a classic model (*METHOD FULL.CPL MODEL.SI*), which can be found in Athena user's book.

Both temperature and duration of diffusion process influence the doping profile. To investigate the influence of temperature annealing, 60 minutes diffusion steps are simulated at 700, 800 and 900°C, the maximum temperature already used for this furnace. Doping profile is present in [Figure 4.24](#). Performing a phosphorous diffusion at 700°C diffusion (black curve) is quite inefficient because it leads to a poor penetration depth of impurities (doping concentration drops below 10^{19} atom/cm³ beyond a 3 nm depth). By increasing the diffusion temperature, the critical diffusion length is increased, to reach almost the full 140nm thickness of the top-Si layer at 900°C (white curve).

In order to investigate the influence of the diffusion time on penetration depth, doping profile is determined for a 15, 30 and 60 minutes diffusion step at 900 °C, corresponding respectively to the white, black and red curves in [Figure 4.25](#). One notices that durations under 60 minutes are not sufficient to reach the desired doping level over the first 100 nm of the top-Si layer. The result after two successive 60 minutes diffusion steps at 900°C is also presented. It permits to reach a slightly higher charge carrier concentration than the one obtained with a single 60 minute diffusion step, especially for the region near the BOX. However, reaching such a level in the present case is not needed. So, **performing a 60 minutes phosphorous diffusion at 900°C is so a good way to ensure to get the desired doping level for electrical characterisation.**

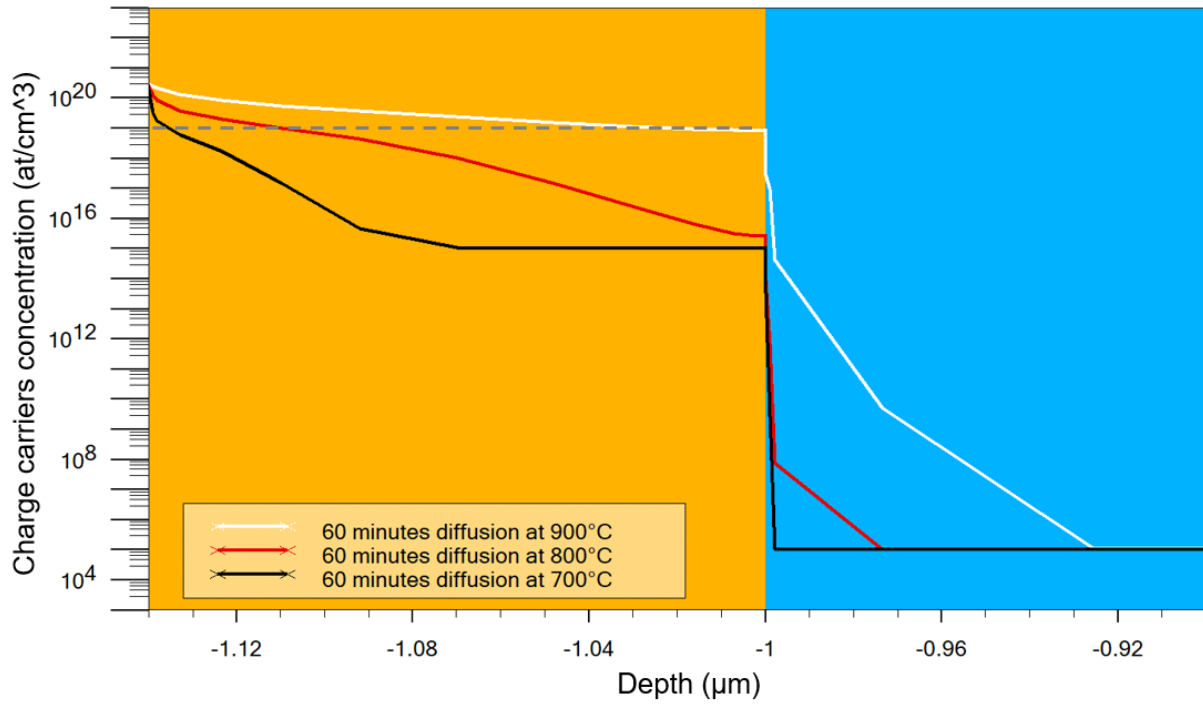


Figure 4.24: Doping profile after a phosphorous diffusion performed at 700, 800 and 900°C during 60 minutes. Orange region represents top-Si, blue one BOX. Doted line defined to desired carrier concentration of the 100 first nanometers of the top-Si layer.

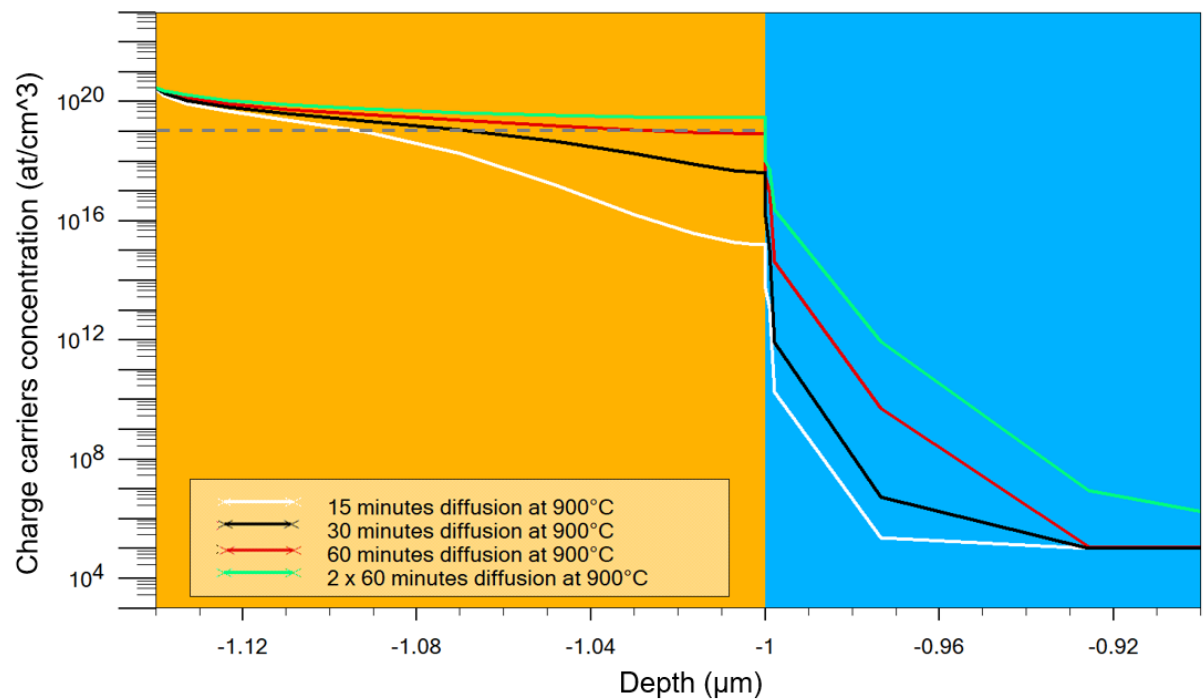


Figure 4.25: Doping profile after a phosphorous diffusion performed at 900°C during 15, 30 and 60 minutes. The influence of successive 60 minutes diffusion steps at 900°C is also presented. Orange region represents top-Si, blue one BOX. Doted line defined to desired carrier concentration of the 100 first nanometers of the top-Si layer.

Chapter 5

Characterisation and discussion

None of the two routes revealing to be successful, the extraction of stress/strain curve with the deduction of opto-electro-mechanical properties of the present silicon specimens is not possible. However, Internal stress associated to each actuator material can be deduced, based on SEM observations of free released actuators.

End of a free released actuator is shown in **Figure 5.1**. Being in the range of small deformation ($\epsilon \leq 0.003$ [22]), the strain mismatch is defined as

$$\epsilon_{mis}^{act} = \frac{u_{free}}{L_{a0}^{free}} \quad (5.1)$$

The free displacement is measured at 4 places for each actuator, two of them are represented by blue en orange arrows on the figure. Same measurements are performed for the lower cursors, and the free displacement is defined as the mean of the four measurements. This permits to take into account a possible misalignment between the photomasks and the existing structures [22].

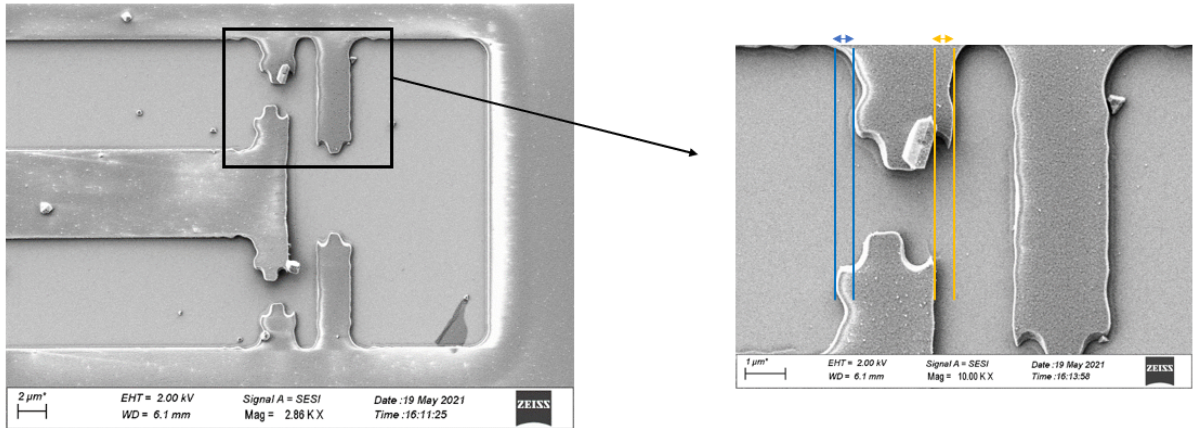


Figure 5.1: End of a free released actuator. A focus is made on the top cursors zone.

For the nickel actuator, a set of 17 free actuators is selected with length from 130 to 1187 μm . Internal stress being an intrinsic property of material, it is not expected to vary with the actuator dimensions. **Figure 5.2** displays the mismatch strain derivation

for different actuator lengths. The variation of internal stress appears to decrease with the increase of actuator length, which is quite surprising. One could have predicted that the reverse trend happens due to the following justification. During the release, an under-etching of silicon oxide located in the neighbourhood of the actuator, beyond the anchoring line, occurs. This increases slightly the real length of released actuator L of a factor ΔL , depending only on the release time and independent of the beam length. By supposing in this proposition that mismatch stress does not vary with the dimensions of the actuator, the relative increase $\Delta L/L$ of the length is more important in small length actuators, so the error on the considered actuator length for a displacement is more important for the small length : one may so under-evaluate the mismatch strain for small length. However, the reverse occurs here.

Mismatch strain can be also deduced from the Stoney measurements, by using the formula

$$\epsilon_{mis}^{act} = \frac{1 - \nu}{E} \sigma_{int} \quad (5.2)$$

Where E and ν are the Young modulus and Poisson ratio of the actuator. Typical Young's modulus and Poisson ratio value for Nickel are 205 GPa and 0.31, this leads to a mismatch strain about 0.00205. Curvature measurement was performed on the test wafer SiN08 the same day due to the creep of nickel. This value is really close to the one (0.00197) obtained for the smallest actuator (130 μm long). Those values are quite lower than the one found in literature (1.16 GPa of biaxial internal stress leading to a mismatch strain of 0.0039 [25]).

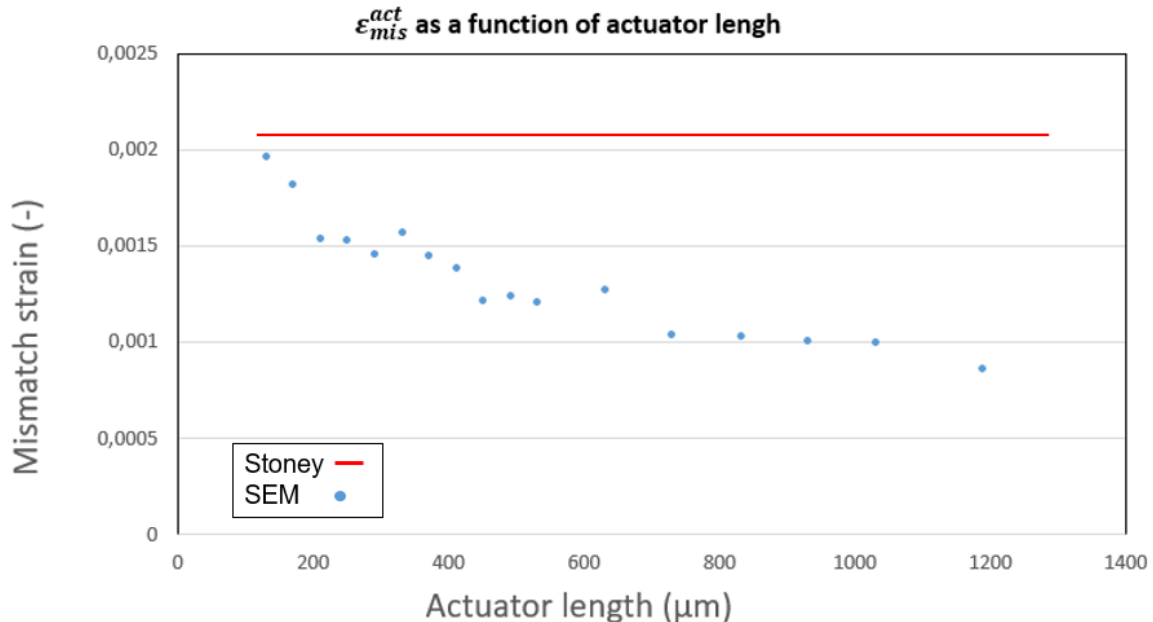


Figure 5.2: Estimated mismatch strain by SEM for different actuator lengths for nickel route. Estimated value with Stoney is also represented

For the PECVD *SiN* route (performed before the *Oxford* chamber cleaning), measurements were performed on five free similar structures, 500 μm long. The mean of those

measurements gives a mismatch strain of 0.00087, equivalent of 186 MPa of internal stress. This is lower than the one obtained through Stoney measurements, for wafer having undergone the same steps (780 MPa leading to a 0.00367 mismatch strain if Young's modulus and Poisson ratio of *SiN* are considered as 155 GPa and 0.27). This discrepancy can maybe come from the values chosen for materials parameters.

Results confirm that the two investigated actuator routes are less effective than the traditional LPCVD silicon nitride one. Maximum stress reached in an annealed PECVD silicon nitride film without cracks apparition is about 1060 MPa. E-beam evaporated nickel induced at maximum 625 MPa of internal stress after deposition, followed by their decrease due to creep. This creep trend was confirmed after two days, by measuring 608 MPa of internal stress. LPCVD *SiN* films owning internal stress higher than 1100 MPa without annealing [8].

Literature relates the change of PECVD *SiN* etch rate in *HF*-49% from 2.3 nm/sec to 12.8 nm/sec, only due to the change of deposition recipe [60]. It has to be notice that the 3 gas flows are different than in our case, and their ratio different (a range from 600 to 900 sccm for *SiH₄*, from 20 to 55 sccm for *NH₃* and from 1200 to 1960 sccm for *N₂*). The PECVD *SiN* recipe developed in this work turned to expose much lower etch rate with the same acid concentration (8 nm/min).

A limitation for the present process is to use *HF*-49% and not *HF*-73%. Indeed, using *HF*-73% permits to reduce the release time, so to limit the etch rate of the actuator. Release steps with this concentration were performed by Urena et al. for 15 μ m width LPCVD *SiN* actuators without noticing significant damage or thickness reduction in the actuators [7]. This could be a solution for the PECVD *SiN* route. Resistance of nickel in *HF*-73% being known to be quite poor, *HF*-49% is preferred if such actuators are concerned.

Chapter 6

Conclusion

This master thesis was devoted to extend the concept of on-chip characterisation developed at UCLouvain to the biaxial loading mode, applied to a silicon thin film. On-chip structures present numerous advantages over other loading testing structures, allowing a multi-physics characterisation of the obtained specimen. Those structures are based on an internal actuation method : the specimen to deform lies on a sacrificial layer. A particular material owning high internal stress, called *actuator*, is deposited and patterned, by ensuring a partial overlap between specimen extremities and actuator. The sacrificial layer is then removed by wet etching. Actuator and specimen becoming in a free-standing state, the internal stress of the actuator can be transmitted to the specimen to deform it. Main advantages of this method are the high throughput capabilities, the possibility to perform statistical analysis, the high reproducibility degree, the absence of calibration due to the internal actuation, and the TEM in-situ observation capability.

The present lab on-chip aimed at characterizing the mechanical properties (like Young's modulus and fracture strain), electrical properties (like piezoresistivity and charge carrier mobility) and optical properties (through transmission measurements). The first step in the lab on-chip conception was to design convenient specimens, leading to biaxial strain levels between 0.001 to 0.04. The way used to sweep different strain levels with internal actuation devices is to vary the components (actuator and specimen) dimensions. In order to have an insight about the ability of each dimension to vary the strain level in the specimen, an analytical model based on the linear elasticity theory was derived, for a simplified system constituted of a square specimen attached to one trapezoidal actuator in each direction. Results of investigation showed that the variation of actuator large basis induces a large modification of strain whereas a variation of actuator length leads to a slighter modification. Then, finite element simulations were performed, for two purposes. Firstly, to derive convenient specimen shapes. Macroscopic biaxial tensile testing of metallic thin sheets used typically cruciform specimens. Simple cruciform specimen has the drawback that once loaded, the strain concentrates at some spots, where fracture has a high probability to initiate. This specimen has to be tuned with some attributes, like rounded region between actuators, fillet disks or slits, in order to decrease the stress at those concentration points and to increase it in the center of the specimen, called gauge region. It appears that situation is improved by performing a thinning of this gauge. Dimensions of attributes are optimised, and the most suited structure designs are selected to be put on the lithography

mask. The second purpose of the FE analysis is to derive a set of component dimensions allowing to sweep the biaxial strain range from 0.001 to 0.04, and to validate the trends emphasized by the analytical results. This trend was validate by FEA and even enhanced to increase the compacity of the structure. Finally, it was decided to include on the mask structures with large actuator to sweep large strain ranges and narrow actuator structures to sweep smaller ranges. Convenient dimensions for both structures style are selected.

The labs on-chip were then fabricated in *WINFAB* facilities. The process is composed of 4 main steps, starting from SOI wafer, to obtain at the end released strained specimen. They are the **wafer selection** (processed wafers are n-doped SOI wafers with a top-silicon layer thickness of 140 nm and BOX of 1 μm), the **specimen creation** consisting in patterning the top-silicon of the SOI wafer to get the desired specimen, the actuator creation, consisting in deposition and patterning the material devoted to transmit its internal stress to the specimen, and the **release** to create the free-standing structures. Due to some technical issues, the traditional actuator material used for lab on-chip, the LPCVD silicon nitride, was not available. Two novel routes was investigated to substitute this missing material : PECVD silicon nitride actuators and nickel actuators. The use of unusual actuator materials, in addition to use of recent *WINFAB* machines, required a non negligible number of operation parameter optimisation steps.

Unfortunately, none of the two routes turned out to reveal fruitful. For nickel actuators specimens, a detachment of specimen an actuator occured, while may be due to the presence of native oxide between the two components, etched during the release. For PECVD silicon nitride actuators, some cracks appeared in the actuators after the thermal annealing aiming at increasing the internal stress. The annealing being performed at low temperature to avoid this problem. The main justification is based on an important cleaning of the PECVD chamber between the optimisation of the recipe and its application on a real SOI wafer. This induces a change of entrance gas flow in the chamber, leading to a film with a different composition. This might cause a strong change of etch rate in $HF - 49\%$. This trend has already been noticed in literature.

The logic following of this work should be this one. A short term potential continuation can be firstly to confirm the hypothesis linked to the cracks apparition in the PECVD silicon nitride. Investigations about an alternative PECVD *SiN* recipe, allowing high internal stress after a suited annealing, can be performed. The actuator etch rate in 49% has also to be carefully determined to avoid encountered problems. A second short term potential solution is to find the exact cause of the presence of native oxide between actuator and specimen overlap. If the problem is linked to the environment in the VACOTEC at the beginning of the pressure drop, a solution can be to subcontract this metallisation to firm with dedicated deposition devices. Long term solutions is to wait until the LPCVD silicon nitride becomes available. Also, etching holes on the ACTUATOR photomask are quite spaced. A solution can be to refabricate this mask with closer holes, to decrease the release time and so limitating the actuator etching.

Strained specimens can then be obtained, one fabrication problems solved, and characterised by SEM measurements and Raman spectroscopy to derive the strain level. In

this way, stress/strain curves can be derived and Young's modulus of the specimen can be obtained. Raman spectroscopy can be used to perform a strain mapping on the specimen. This mapping can be compared to the FEA results. Fracture strain of specimen can also be extracted and survival probability can be deduced.

Once these mechanical results obtained, more complex processes involving additional fabrication steps can be performed to build labs on-chip with other functional proposes. Among them, one can build lab on-chip devoted to Raman transmission measurements or TEM observation. A back etching step is thus needed. Other labs on-chips devoted to electric properties characterisation can be fabricated, inducing the deposition of electric tracks and annealing to create an ohmic contact between silicon and metallic track. Probing measurements can be performed to extract informations about piezoresistivity. The final aim of such lab on-chip is the creation of PIN homojunctions, where the intrinsic region is under strain. Such device can constitutes a novel route in the development of photodetector working in the SWIR wavelength region. All these structures being envisaged in the conception phase, photolithography mask associated to each step is already available.

This master thesis constitutes an efficient way to learn, develop and reinforce existing knowledges linked to several domains of physics of materials. In addition to theoretical hard knowledge linked to material science, mechanics and optoelectronics, soft knowledges were also developed like the use of softwares, like *COMSOL* for FE simulations, *SILVACO* for doping modelling and more classically *MATLAB* and *EXCEL* for data treatment. *WINFAB* fabrication permitted to acquire practically knowledges, and to develop the judgemental analysis of the obtained results. In a sense, the breakdown of the LPCVD device can be view as a chance : this paved the way to unusual routes, permitting to go off the beaten tracks and not to systematically apply a well elaborated process. Even if the initial aim of this master thesis, obtaining biaxially strained structures, was not achieve, the optimisation steps like anisotropic RIE of silicon, PECVD of silicon nitride or thick chromium layer deposition can be useful to other researchers needing to perform similar steps.

Appendices

Design

Derivation of effective width for a trapezoidal actuator

The hypothesis based on the present derivation of the effective width is to consider the actuator as the connection in series of elements with infinitesimal width $w(x)$. To perform such derivation, one has to use the general relation between the effective stiffness constant k_{eff} and the constant associated to each discrete element k_i , and the relationship between stiffness constant and Young's modulus for a regular rod of length L , cross section A (and displacement u) :

$$\Delta L = \frac{F}{k_{eff}} = \sum_i \Delta L_i = \sum_i \frac{F}{k_i} = F \sum_i \frac{1}{k_i} \quad \rightarrow \quad \frac{1}{k_{eff}} = \sum_i \frac{1}{k_i} \quad (6.1)$$

$$\sigma = \frac{F}{A} = \frac{k}{A} u \quad \text{and} \quad \sigma = \epsilon E = \frac{u}{L} E \quad \rightarrow \quad \frac{1}{k} = \frac{L}{A E} \quad (6.2)$$

Remembering that the cross section can be generally expressed as $A(x) = t w(x)$ with t the constant thickness, the effective stiffness constant can be obtained by integrating the width $w(x)$ over the full length of the actuator L (here $w(x) = w_0 + (w_{max} - w_0)\frac{x}{L}$) :

$$\frac{1}{k_{eff}} = \sum_i \frac{\Delta x}{t w(x) E} = \frac{1}{E t} \int_0^L \frac{1}{w(x)} dx = \frac{L}{E t} \frac{\ln\left(\frac{w_{max}}{w_0}\right)}{w_{max} - w_0} \quad (6.3)$$

This makes appear the effective width w_{eff} :

$$k_{eff} = \frac{E t}{L} w_{eff} \quad \text{with} \quad w_{eff} = \frac{w_{max} - w_0}{\ln\left(\frac{w_{max}}{w_0}\right)} \quad (6.4)$$

General behaviour lines graph

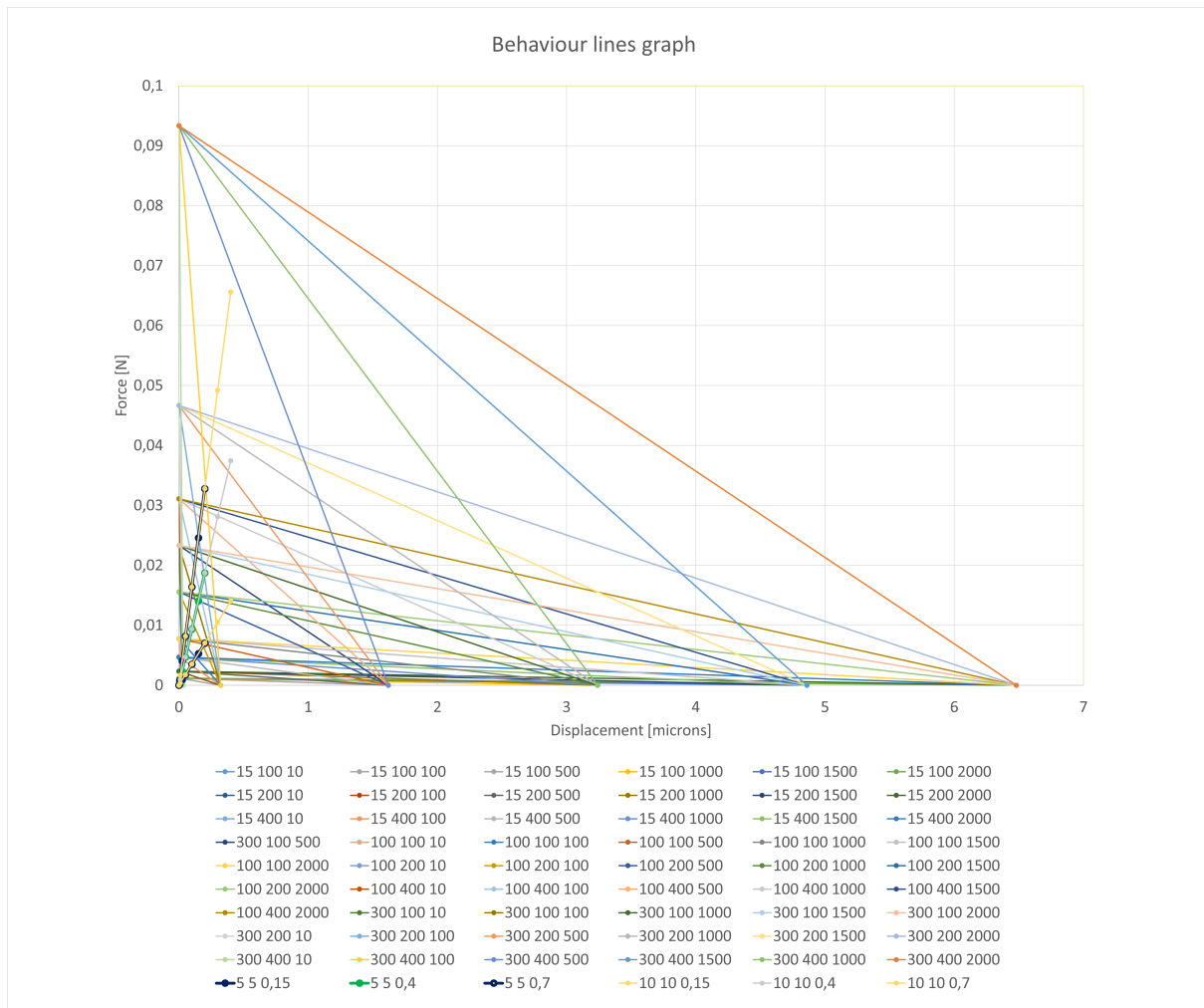


Figure 6.1: General behaviour lines graph

Influence of gauge region shape

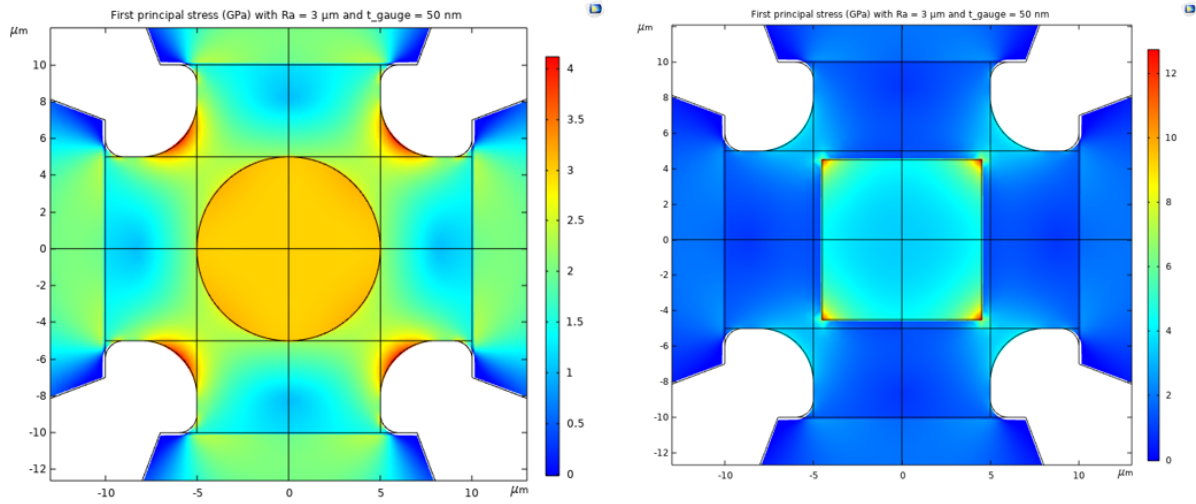


Figure 6.2: Shape of the gauge region, thinned to 50 nm. High hotspots are present in the square one, due to the corner. Circular one is so the most suited

First principal stress

In order to understand the meaning of the first principal stress, we have to make a reminder about Mohr's circle. Let us represent an arbitrary point P of a structure by a cube placed in a coordinate system (each of the axis being parallel to the normal of a face so $\hat{e}_x$, $\hat{e}_y$ and $\hat{e}_z$) and let us apply to it a plane stress (in the xy plane). Without any hydrostatic stress and contact with other bodies, the stress tensor of the system can be expressed with only 3 independent components in the actual coordinates system : the stress along $\hat{e}_x$ applied on the square edge of normal $\hat{e}_x$, the stress applied on the second edge of the square parallel to its normal $\hat{e}_y$ and the shear stress applied on the face of normal $\hat{e}_z$:

$$\underline{\underline{\sigma}} = \begin{pmatrix} \sigma_{xx} & \tau_{xy} & 0 \\ \tau_{yx} & \sigma_{yy} & 0 \\ 0 & 0 & 0 \end{pmatrix} \quad (6.5)$$

By performing rotations of the initial frame of reference, one can find a particular axis orientation in which stress can only be expressed by normal stress component (called *principal stresses*) without any shear component. Mohr's circle is a geometrical representation which allows to find these *principal stresses*, their associated orientations and the maximum shear stress in each plane.

Let us first focus on the xy-plane (Figure 6.3, left). One can define a unit vector $\vec{n}$ in this plane with an arbitrary direction. Stress applied to the faces of the cube can be expressed by a component parallel to $\vec{n}$ (called σ_n) and a component normal to $\vec{n}$ (called τ_n). We can sweep a 360° angle sector in the xy plane (starting from the initial arbitrary direction) and for each particular angle evaluate the value of the two components (σ_n, τ_n) defined previously. These couple forms in the (σ_n, τ_n) graph a circle called **Mohr's circle**. Two opposite point in the circle represents a particular orientation of the $\vec{n}$ vector : one point has the coordinates (σ_{xx}, τ_{xy}) and the second point ($\sigma_{yy}, -\tau_{xy}$). If θ is defined as the angle between $\vec{n}$ and $\hat{e}_x$, the angle formed between each point and the horizontal axis in Mohr's circle is 2θ ¹. This circle can so be constructed for the 3 planes containing each one two of the three principal stress. Mohr's circle for general plane stress situation is represented in (Figure 6.3, right). By considering that $\sigma_I \geq \sigma_{II}$, the maximum shear stress present in the system is located in the

By using stresses in the referential axis system (σ_{xx}, σ_{yy} and τ_{xy}), principal stresses can be expressed based on geometrical analysis :

$$\sigma_I = \frac{\sigma_{xx} + \sigma_{yy}}{2} + \sqrt{\frac{\sigma_{xx} - \sigma_{yy}}{2}^2 + \tau_{xy}^2} \quad (6.6)$$

$$\epsilon_I = \frac{\epsilon_{xx} + \epsilon_{yy}}{2} + \sqrt{\frac{\epsilon_{xx} - \epsilon_{yy}}{2}^2 + \left(\frac{\gamma_{xy}}{2}\right)^2} \quad (6.7)$$

¹This comes from that fact that the surface normal to $\vec{n}$ is divided by a factor $\cos\theta$ and each of the force components normal to $\vec{n}$ is multiplied by a factor $\sin\theta$, leading after simplification a tangential component τ_{xy} proportional to the stress σ_0 (for $\vec{n} = \hat{e}_x$) by a factor depending on $\sin 2\theta$.

Mohr's circle in the case of plane stress

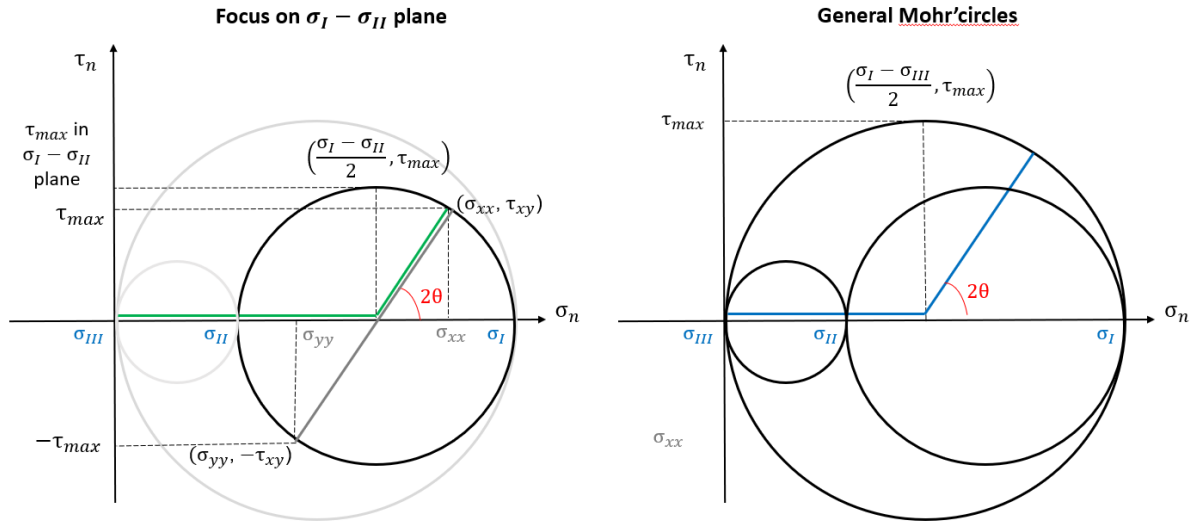


Figure 6.3: Schematic representation of Mohr's circle for the present situation ($\sigma_{xx} =$)

Principal directions can be derived (based on continuum mechanics analysis), such that :

$$\tan 2\theta = \frac{2\tau_{xy}}{\sigma_{xx} - \sigma_{yy}} \quad (6.8)$$

Convergence studies for FEA

Convergence study for slitted structure

Firstly, a range from $2\ \mu\text{m}$ to $50\ \mu\text{m}$ is swept for the maximum element size parameter, and the minimum size element parameter is set to the maximum one divided by 100. The probed stress is the general stress present in the structure, located in the corner of a slit. In [Figure 6.4 \(left\)](#), if the error threshold is arbitrarily defined as 10 MPa, the larger possible mesh size element is about $21\ \mu\text{m}$. Given that the same element size parameters are selected for further investigations, taking a value a bit under this value ($15\ \mu\text{m}$ e.g.) provides kind of security. To get a convenient computational time, a second parametric sweep is performed on the minimum element size only from 1 nm to $1\ \mu\text{m}$. Graph is shown on right. If one defines arbitrarily an error tolerance threshold of 1%, the situation is convergent below a minimum size element of $0.17\ \mu\text{m}$. So, taking maximum and minimum element size of $15\ \mu\text{m}$ and $150\ \text{nm}$ respectively seems to constitute a good compromised between mesh-independency and time computation.

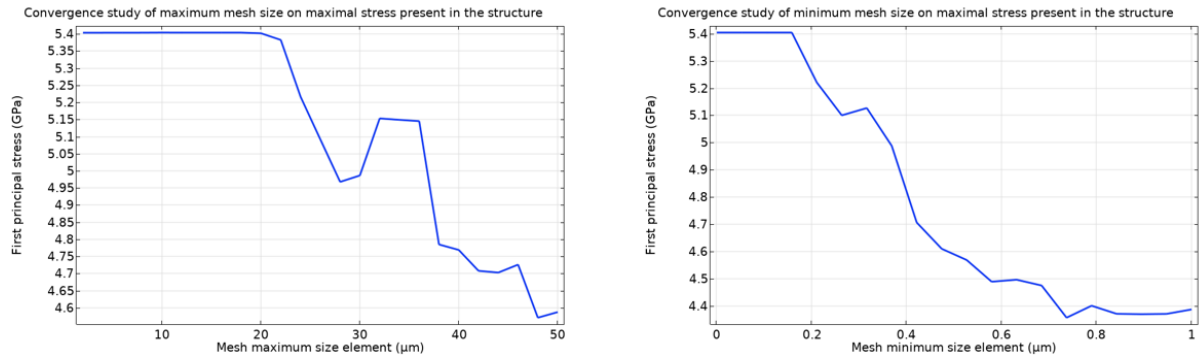


Figure 6.4: Convergence study for slitted structure : first principal stress against maximum element size (left), and against minimum element size for maximum one set to $15\ \mu\text{m}$ (right).

Convergence study for slitted structure with rounded extremities

[Figure 6.5](#) shows convergence study for structures with rounded extremities slits (minimum size element is set to 100 smaller than the maximum one). If error threshold is set to 10 MPa, a the bigger maximum size element acceptable is $15\ \mu\text{m}$. By setting this value as the maximum one, the convergence study on the minimum size element value reveals, by keeping the same error threshold, that $0.2\ \mu\text{m}$ is the maximum convenient value.

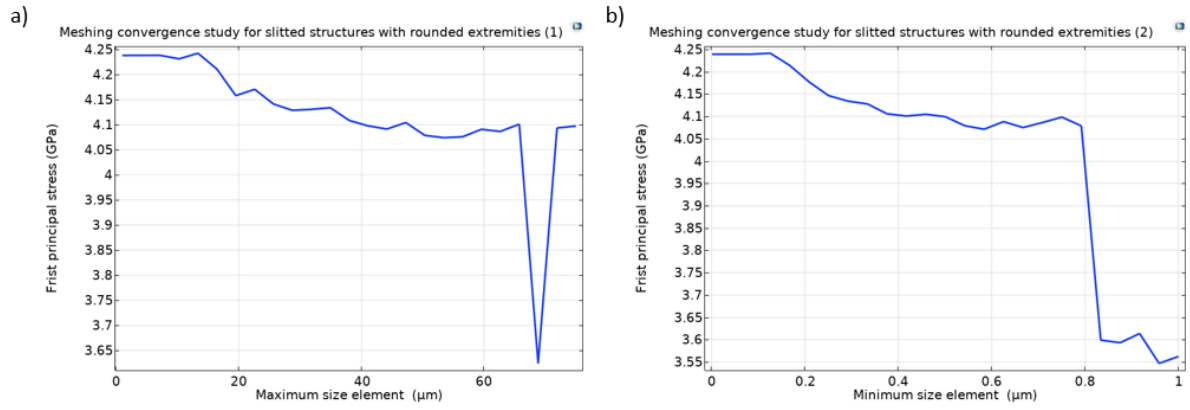


Figure 6.5: Convergence study for slitted structures with rounded extremities

Convergence study for slitted structure with broadened connectors

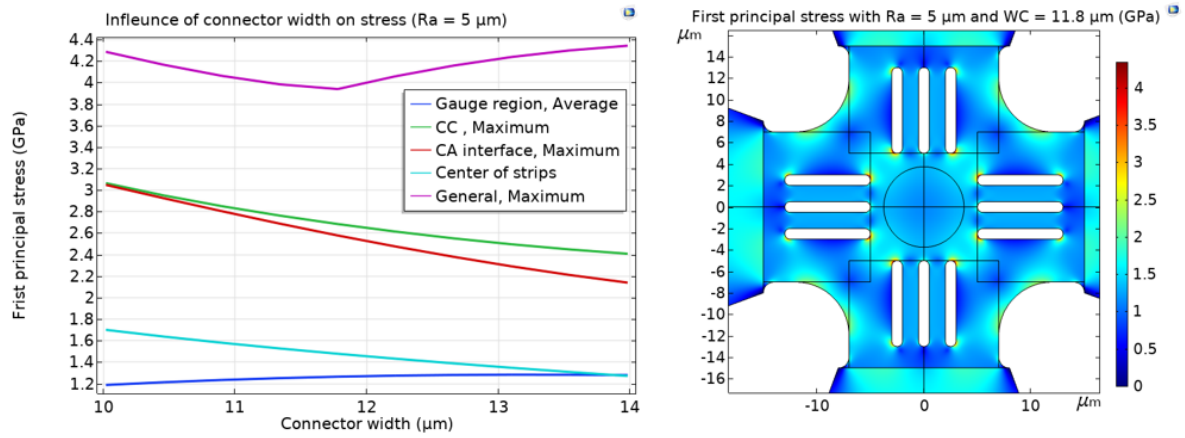


Figure 6.6: Convergence study for slitted structures with broadened connectors. For the second investigation, maximum element size is set to 15 μm. Error threshold set at 10 MPa

Convergence study for basic alternative 2-Actuators structure

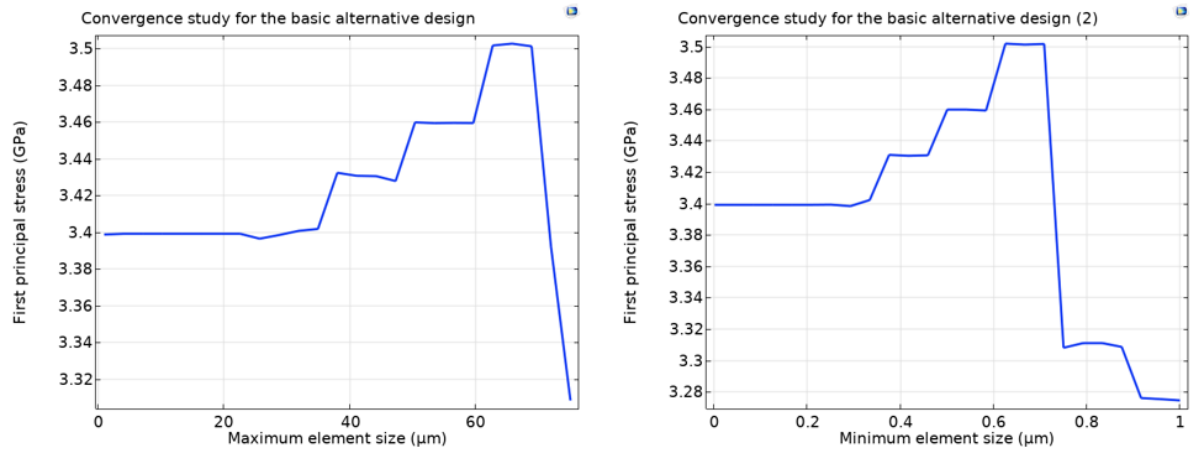


Figure 6.7: Convergence study for basic alternative structure. For the second investigation, maximum element size is set to 25 μm . Error threshold set at 10 MPa

Convergence study for slitted alternative 2-Actuators structure

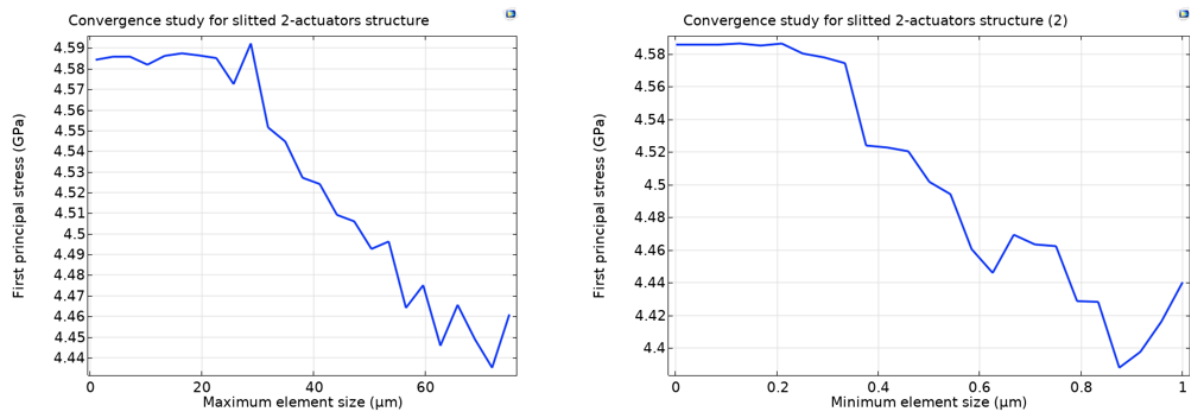


Figure 6.8: Convergence study for slitted alternative structure. For the second investigation, maximum element size is set to 20 μm . Error threshold set at 10 MPa

Schematic representation of elliptical slits

Complete results of simulations

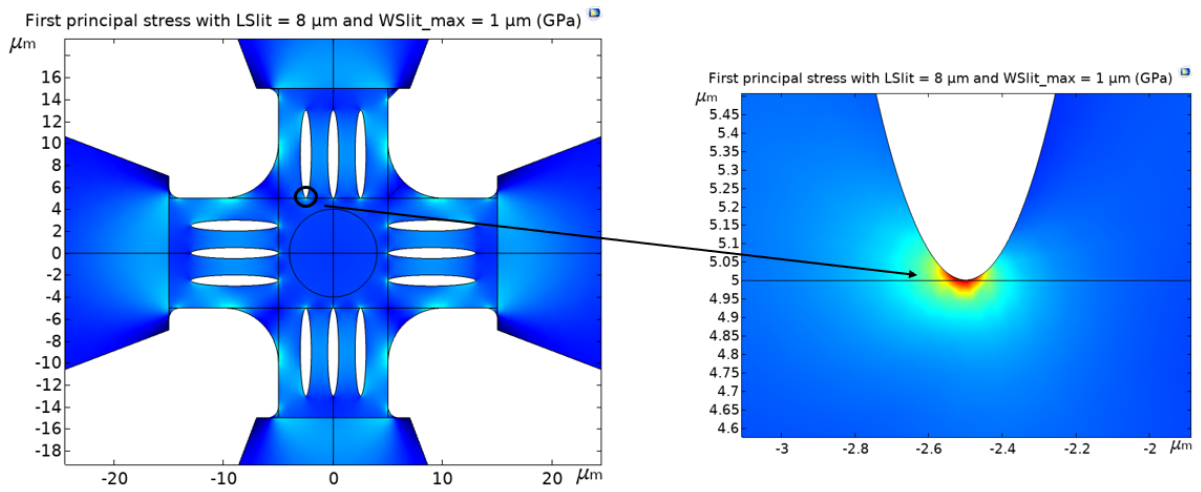


Figure 6.9: Schematic representation of elliptical slits, with $LSlit = 8 \mu\text{m}$ and maximal $WSlit = 1 \mu\text{m}$. Focus of the hotspot is present on right

	Initial case	Rounded region with $R_a = 3\mu m$	Filet disks with $R_u = 2.5\mu m$ (centered)	Filet disks with $R_u = 2.5\mu m$ and $P = 1\mu m$ (shifted)
Gauge region, average (GPa)	1.79	1.14	1.87	1.47
Gauge region, standart dev. (GPa)	0.07	0.039	0.22	0.057
ratio std. dev/average (gauge) (-)	4%	3.4%	11%	3.9 %
Center of connectors (or central strip) (GPa)	2.08	1.27	1.95	1.67
Ratio center of connector/average (gauge) (-)	1.15	1.05	1.04	1.13
Interface C/A (GPa)	3.2	3.69	1.83	2.12
ratio interface C/A / average (gauge) (-)	1.78	3.2	0.98	1.44
Highest F.P.Stress value (GPa)	6.61	3.69	3.94	3.39
Highest F.P.Stress value/average (gauge) (-)	3.69	3.2	2.11	2.3
Highest F.P.Stress location	corner CC	interface CA	Filet proximity	Filet proximity

Table 6.1: Main results of simulations for specimen with 4 actuators without slits. Third column refers to the design obtained in the first investigation, fourth and fifth ones to the second one.

	Initial case	Rectangular Right angle $L\text{Slit} = 8 \mu\text{m}$ $W\text{Slit} = 1 \mu\text{m}$ $CW = 10\mu\text{m}$	Rectangular Rounded extremities $L\text{Slit} = 8 \mu\text{m}$ $W\text{Slit} = 1 \mu\text{m}$ $CW = 10\mu\text{m}$	Elliptical $LD = 8 \mu\text{m}$ $SD = 1 \mu\text{m}$ $CW = 10\mu\text{m}$	Rectangular Widened $L\text{Slit} = 8 \mu\text{m}$ $W\text{Slit} = 1 \mu\text{m}$ $CW = 11.8\mu\text{m}$
Gauge region, average (GPa)	1.79	1.20	1.17	1.18	1.27
Gauge region, standart dev. (GPa)	0.07	0.031	0.022	0.019	0.054
ratio std. dev/average (gauge) (-)	4%	2.6 %	1.9%	1.7 %	5.5 %
Center of connectors (or central strip) (GPa)	2.08	1.78	1.68	1.73	1.48
Ratio center of connector/average (gauge) (-)	1.15	1.48	1.43	1.47	1.17
Interface C/A (GPa)	3.2	2.89	2.96	2.88	2.57
ratio interface C/A / average (gauge) (-)	1.78	2.41	2.53	2.44	2.03
Connector/connector rounded region (GPa)	6.61	3.04	3.03	2.83	2.68
ratio CC (-)	3.69	1.78	1.68	1.73	2.11
Highest F.P.Stress value (GPa)	6.61	5.4	4.25	7.12	3.95
Highest F.P.Stress value/average (gauge) (-)	3.69	4.5	3.63	6.03	3.11
Highest F.P.Stress location	corner CC	Slit extremity	Slit extremity	Slit extremity	Slit extremity

Table 6.2: Main results of simulations for structures with 4 actuators with slits. Third column of results refers to slits with right angle at corners, fourth one to slits with rounded angles with a radius $Rsr = 0.5 \mu\text{m}$, fifth one to elliptical slits and sixth one to slitted specimens with widened connectors.

	Rounded region with $R_a = 5\mu m$	Slits with $R_a = 5\mu m$ $LSlit = 8\mu m$ $WSlit = 1\mu m$ $CW = 10\mu m$	Slits with $R_a = 5\mu m$ $LSlit = 8\mu m$ $WSlit = 1\mu m$ $CW = 14\mu m$	Filet disks with $R_u = 2.5\mu m$ (centered)	Filet disks with $R_u = 2.5\mu m$ (shifted) with $P = 1\mu m$
Gauge region, average (GPa)	0.9	0.85	0.93	1.43	1.29
Gauge region, standard dev. (GPa)	0.043	0.038	0.057	0.306	0.209
ratio std. dev/average (gauge) (-)	4.8 %	4.5 %	6.2 %	21.4 %	16.2 %
Center of connectors (or central strip) (GPa)	1.07	1.23	0.94	2.6	2.47
Ratio center of connector/average (gauge) (-)	1.19	1.44	1.01	1.81	1.91
Interface C/A (GPa)	3.34	2.75	2.35	2.99	3.52
ratio interface C/A / average (gauge) (-)	3.71	3.24	2.53	2.09	2.73
Connector/connector rounded region (GPa)	2.64	2.35	1.90	5.23	4.94
ratio CC (-)	2.93	2.76	2.04	3.66	3.83
Highest F.P.Stress value (GPa)	3.34	4.59	4.76	5.23	4.94
Highest F.P.Stress value/average (gauge) (-)	3.71	5.4	5.11	3.66	3.83
Highest F.P.Stress location	Interface CA	Slit extremity	Slit extremity	Interface CA	Interface CA

Table 6.3: Main results of simulations for specimens with 2 actuators. Second column refers to specimen with rounded region centered at the right angle between connectors, Third column the specimen with slits (with rounded slits corners), fourth the specimen with centered filet disks and fifth with ex-centered filet disks.

Fabrication process

Effect of thermal annealing on *PECVD SiN* internal stress

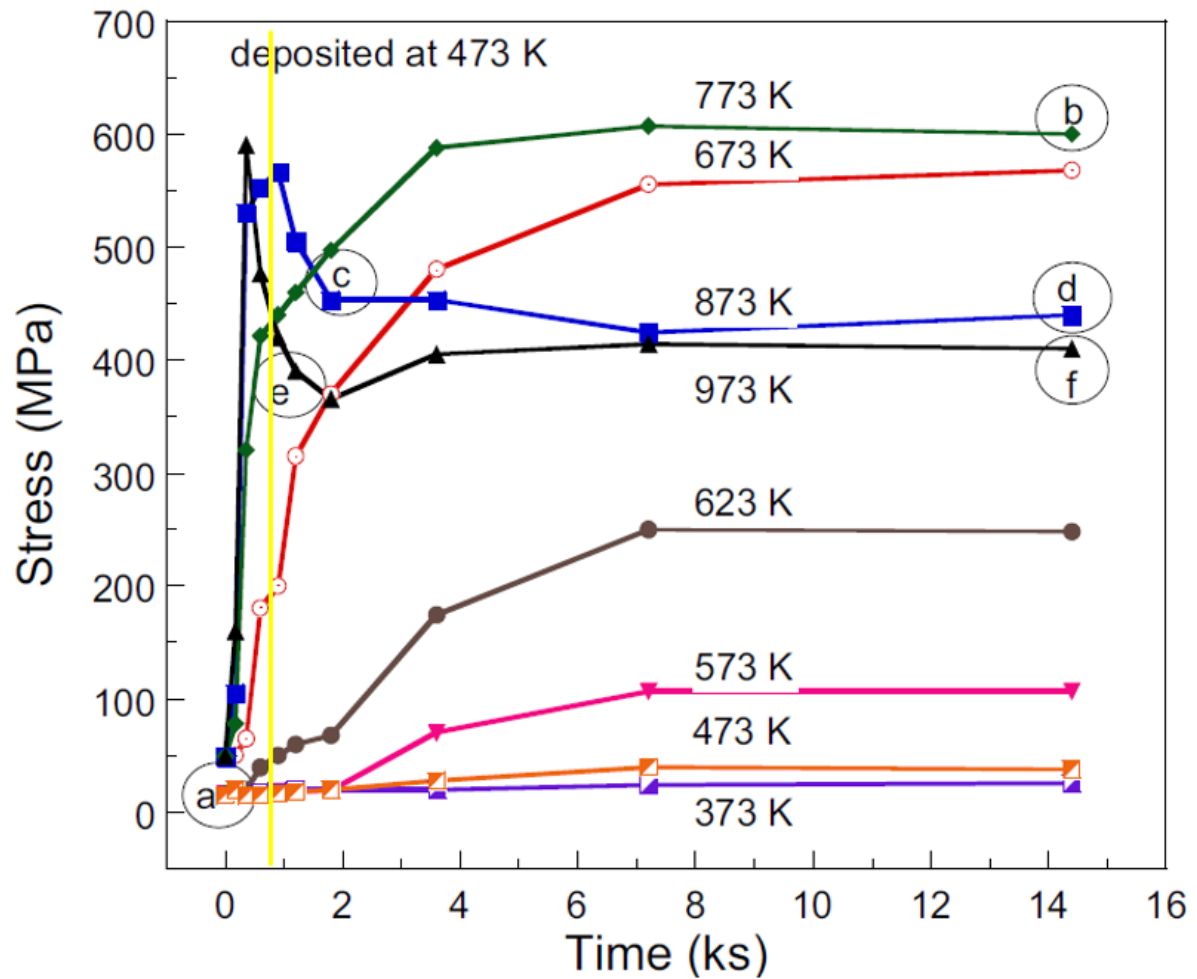


Figure 6.10: Effect of thermal annealing at different temperatures and durations. Yellow vertical line correspond to a 15 minutes annealing.[54]

Process sheet dedicated to the 140nm-thick specimens

Process biaxial			SUL 5012	SUL 5015 (Ni)	SUL 5016(SiN)	SiN07 (wafer test SiN)	SiN06 (wafer test Ni)	
STEP 1 : Wafer selection								
#1	Measurement palmer	Top-Si BOX				385/385/387 μm	385/385/385 μm	
#2	Measurement Dektak					- 3045 nm	- 2499 nm	
#3	Measurement ellipsometer		145 nm	142.5 nm	145 nm			
			1002 nm	996 nm	997 nm			
#4	Edge smoothing with sandpaper (SiC 100 then 150 nm)							
STEP 2 : Specimen creation								
#5	Cleaning standard (with HF)	Mask2020 - SPECIMEN - 140 mJ/cm^2						
#6	Lithography Positive							
#7	Check microscope		Poor	Nice except lower instance				
#8	Silicon plasma etching (Corial, CHF3/SF6)		45 sccm CHF3/12 sccm SF6, 21 seconds					
#9	Check microscope							
#10	Photoresist wet stripping							
#11	Cleaning standard (with HF)							
#12	Lithography Positive	Mask2020 - THINNING - 160 mJ/cm^2						
#13	Check microscope			Nice				
#14	Silicon plasma etching (Corial, CHF3/SF6)			50 sccm CHF3/5 sccm SF6, 10 seconds				
#15	Check microscope							
#16	Measurement ellipsometer	110 nm			118 nm			
		100 nm			105 nm			
		998 nm		997 nm				
#17	Photoresist wet stripping	Top-Si (center) Top-Si (extremity) BOX						

Process biaxial			SUL 5012	SUL 5015 (Ni)	SUL 5016(SiN)	SIN07 (wafer test SiN)	SIN06 (wafer test Ni)
STEP 3 : Actuator creation							
#18	Cleaning standard (with HF)	DRY950 CMOS 27 nm					
#19	Thermal oxydation (KOYO)						
#20	Lithography Positive		Mask2020 - WINDOW OPENING - 150 mJ/cm^2				
#21	Check microscope			Nice			
#22	Etching SiO2 thermal wet BHF		30 seconds				
#23	Check ellipsometry		SiO2	No SiO2 remaining at opennings		24 nm	24 nm
#24	Photoresist wet stripping						
#25	Cleaning standard						
#26	Deposition Si3N4 PECVD (SiH4/Nh3/N2)		1000 sccm SiH4/ 21 sccm NH3/ 1500 sccm N2, 20 minutes				
#27	Measurement Dektak					- 9667 nm	- 2528 nm
#28	Measurement ellipsometer	Si3N4			453 nm		
#29	Thermal annealing TEMPRESS	600°C, 15 minutes, 0.5 l/min N2					
#30	Measurement Dektak				-39 289 nm		
#31	Check microscope		Small cracks		Small cracks		
#32	Cleaning standard (without HF)						
#33	LP3						
#34	Lithography Positive	Mask2020 - ACTUATOR - 160 mJ/cm^2					
#35	Check microscope		Nice				
#36	Si3N4 PECVD plasma etching (OXFORD, CHF3/02)	100 sscm CHF3/10 sccm O2					
#37	Check microscope		Nice				
#38	Measurement ellipsometer						
#39	Lithography negative	Mask2020 - ACTUATOR - 120 mJ/cm^2					
#40	Cleaning standard (with HF)						
#41	Deposition Nickel VACOTEC						
#42	Lift off						
#43	Measurement Dektak					-18 273.6 nm	
#44	Photoresist wet stripping						
STEP 4 : Realease							
#45	HF Release						
#46	Check microscope						
#47	CPD						

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École polytechnique de Louvain

Rue Archimède, 1 bte L6.11.01, 1348 Louvain-la-Neuve, Belgique | www.uclouvain.be/epl