

Study of the ferromagnetic resonance in arrays of magnetic nanowires for filtering applications

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Abstract

This work aims to study the ferromagnetic resonance absorption of microwave electromagnetic signals using ferromagnetic nanowires grown in nanoporous alumina membranes. Two different types of NiCo nanowires with more nickel or more cobalt content are compared on the basis of their magnetic properties and their filtering capabilities. Specific characteristics of the materials are extracted as well as general conclusions. Particularly, an absorption of more than 99% is shown using high nickel content NiCo nanowires. The fabrication process of a microwave filter using a substrate integrated waveguide with ferromagnetic nanowires as filtering materials is discussed and the influence of the dimensions on the transmission is shown using finite-element analysis software and analytical developments. Finally, some experimental considerations for the general growing of nanowires in anodized alumina membranes and for the specific presented materials and processes are highlighted.

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List of symbols and abbreviations

List of frequently used symbols

Symbol	Unit (SI)	Property
B	T	Magnetic field
E	$N \cdot C^{-1}$	Electric field
f	Hz	Frequency
f_{FMR}	Hz	Ferromagnetic resonance frequency
$f_{c_{mn}}$	Hz	Cutoff frequency of propagation mode TE_{mn} or TM_{mn}
H	$A \cdot m^{-1}$	Magnetic field strength
M	$A \cdot m^{-1}$	Magnetization
M_s	$A \cdot m^{-1}$	Saturation magnetization
Z	Ω	Impedance
α	-	Gilbert damping constant
γ	$GHz \cdot T^{-1}$	Gyromagnetic ratio
γ	m^{-1}	Propagation constant
ϵ	$F \cdot m^{-1}$	Absolute permittivity
ϵ_0	$F \cdot m^{-1}$	Permittivity of free space
ϵ_r	-	Relative permittivity
ϵ_e	-	Effective permittivity
μ	$H \cdot m^{-1}$	Permeability
μ_0	$H \cdot m^{-1}$	Permeability of free space
μ_r	-	Relative permeability
σ	$S \cdot m^{-1}$	Electrical conductivity
χ	-	Magnetic susceptibility
ω	$rad \cdot s^{-1}$	Angular frequency
ω_L	$rad \cdot s^{-1}$	Larmor frequency
ω_{FMR}	$rad \cdot s^{-1}$	Ferromagnetic resonance angular frequency

Abbreviations

AFD	Anisotropy field distribution
AGM	Alternating gradient magnetometer
ccp	Cubic closed packing
DC	Direct current
ECD	Electrochemical deposition
EDX	Energy dispersive X-ray analysis
EM	Electromagnetic
fcc	Faced centered cubic
FMR	Ferromagnetic resonance
hcp	Hexagonal close packing
PMMA	Poly(methyl methacrylate)
RF	Radio frequency
RRR	Residual resistivity ratio
SEM	Scanning electron microscope
SIW	Substrate integrated waveguide
SM	Membranes fabricated by SmartMembranes
Sy	Membranes fabricated by Synkera
TE	Transverse electrical
TM	Transverse magnetic
TEM	Transverse electromagnetic

Chapter 1

Introduction

Magnetic nanowires have recently drawn a particular attention in the field of nanotechnologies thanks to their remarkable properties inherent to their specific shape and thanks to their easy fabrication. They have specifically shown to be adapted for applications in the field of spintronics and microwaves devices [1]. Indeed, the possibility to grow multilayered magnetic nanowires is particularly interesting for the realisation of magnetoresistive devices. On the other hand, the magnetization observed without any applied magnetic field due to the strong anisotropies of nanowires naturally oscillates at frequencies in the range of the microwaves. It is this last property that will interest us in the present work.

The oscillation frequency of magnetic moments can absorb electromagnetic waves oscillating at the same frequency due to the so-called ferromagnetic resonance. This effect can then be used to filter specific frequencies of electromagnetic signals in order to remove for example a noisy frequency. However, in order for this effect to be used, the ferromagnetic materials have to be embedded in a structure propagating electromagnetic waves like a waveguide. Microwave filters are of primary importance in communication systems operating at microwave frequencies such as satellite communications, telephone networks or television broadcasting [2]. In a context of constant improvements of performances and device miniaturization of electronic components, such improvements are also sought for the microwave filters.

Due to their small size and spontaneous strong magnetization, ferromagnetic nanowires can be embedded in so-called substrate integrated waveguides to filter electromagnetic waves. The nanoporous substrates used to grow the nanowires can then be used as support for the waveguide itself. This is particularly advantageous compared to the ferrites usually used for this kind of applications that show no or much weaker spontaneous magnetization at zero applied field. Hence, they need the support of a macroscopic magnet to allow ferromagnetic resonance absorption. Therefore, nanowires with strong spontaneous magnetization embedded in a substrate integrated waveguide could offer a great improvement in terms of miniaturization.

The goal of this work is to describe the filtering properties of ferromagnetic nanowires grown in nanoporous alumina membranes and to design substrate integrated waveguides using ferromagnetic nanowires as band-stop filters.

A theoretical description of the magnetism, and in particular the ferromagnetism and the ferromagnetic resonance will be presented, with a focus on nanowires. Then, the electromagnetic theory of the rectangular waveguides will be developed. The substrate integrated waveguide and the microstrip lines will be presented as two structures allowing the propagation of electromagnetic waves.

Then, the techniques used to fabricate nanoporous membranes and to grow nanowires as well as the different instruments and methods needed to fabricate the desired microwave devices will be explained.

Afterwards, two derivatives of the same ferromagnetic nanowires will be introduced and their properties will be examined in order to characterize their magnetic properties and their filtering capabilities. The important characteristics for a good filtering material are identified and an absorption of more than 99% is shown.

Next, the fabrication process of a substrate integrated waveguide with ferromagnetic nanowires as filtering materials presented in [3] will be discussed and its dimensions will be optimized for the specific case of NiFe nanowires, paving the way for the realization of an experimental realization.

Finally, some experimental tips extracted from the issues encountered during the work will be detailed.

Chapter 2

Theory

This chapter aims to present the main theoretical concepts needed for a complete understanding of the following report. First, the magnetism will be introduced with an emphasis on ferromagnetism and the related phenomena in bulk materials and in nanowires. Secondly, the electromagnetic aspects will be introduced with a focus on rectangular waveguide and microstrip lines.

2.1 Magnetism

This section is mainly based on the books *Magnetism and Magnetic Materials* by J.M.D. Coey [4] and *Introduction to Solid State Physics* by C. Kittel [5] as well as the theses from G. Hamoire [6], J. De La Torre Medina [7], A. Saib [8], M. Darques [9] and the master thesis of V. Van Kerckhoven [10].

Magnetic properties of materials were already used millennia ago. Indeed, naturally magnetized magnets were already found under the form of magnetite magnetized by huge electric currents in lightning strikes. The first known applications of these magnets was for the compass in which a small magnetic material interacts with the magnetic field of the Earth to indicate the North [4]. Today, magnetism is used in a wide range of materials and technologies, ranging from magnets to stick on a refrigerator to electric motors, computer memories or even levitation trains.

2.1.1 The different types of magnetism

The magnetic properties of materials are directly linked to quantum mechanics. Indeed, the origin of magnetism in a material is the magnetic moments of its atoms. Two sources of this magnetic moment can be identified: the spin of the electrons and their orbital angular momentum around the nucleus. All atoms show a tendency to be repelled by a magnetic field since their charges will act in such a way that it shields the interior of the body from the magnetic field. This effect is named **diamagnetism** and is caused by a change in the orbital moment induced by the magnetic field. However, when some electrons of an atom are unpaired i.e. some shells are only partially filled, the **paramagnetic** effect is dominant, which means that the magnetic moment of the atoms will tend to align with the magnetic field. This happens because, due to the Pauli principle, if the electronic shells are not completely filled, a net magnetization arises. Hence, diamagnetism can only be observed for atoms with all filled electron shells. In this case, the atoms have globally zero spin and zero orbital moment, leading to the repulsive effect when a magnetic field is applied.

Some particular paramagnetic materials can retain a magnetization state even without any applied magnetic field. This is possible if the magnetic moments are ordered in arrays. This ordering can only appear under a critical temperature called Curie Temperature. The effects of this ordering on the magnetic moments can lead to three special cases of paramagnetism: **ferromagnetism**, **antiferromagnetism** and **ferrimagnetism**. Ferromagnetic materials show a spontaneous magnetization without any applied magnetic field because their magnetic moments

have some naturally preferred orientations. However, in large ferromagnetic materials composed of multiple magnetic domains, the global magnetization is generally close to zero. Hence, when a magnetic field is applied, all magnetic moments align with the magnetic field. When the magnetic field is then removed, the material keeps its magnetization. Ferrimagnetic materials are similar to ferromagnetic materials but with a smaller remanent magnetization caused by the antiparallel alignment of the magnetic moments. Antiferromagnets are particular ferrimagnetic materials where the magnetic moments are antiparallel and of same amplitude, leading to a globally zero magnetization.

2.1.2 Mathematical description

In vacuum

Let's now mathematically describe the magnetic properties of materials. The magnetic field is usually denoted B and is expressed in *tesla* in the International System of Units (SI). In vacuum, it is linked to the magnetic field strength or H-field H expressed in *ampere per meter* by the relation

$$B = \mu_0 H \quad (2.1)$$

with μ_0 the permeability of free space.

In materials

The H-field is the sum of two contributions

$$H = H_c + H_m \quad (2.2)$$

where H_c is created by conduction currents and H_m is created by the magnetization distributions of other magnets and of the magnet itself. The contribution H_m inside a magnet is called demagnetizing field.

In a material medium, H and B are linked with the magnetization of a magnet M by the following relation:

$$B = \mu_0(H + M) \quad (2.3)$$

The magnetic susceptibility of a material χ describes its magnetic reaction toward the applied field and is defined as

$$\chi = \frac{M}{H} \quad (2.4)$$

We can now link the magnetic susceptibility and the different magnetic phenomena described before. When the susceptibility is negative, the magnetization is opposite to the direction of the applied magnetic field. This means that the material is diamagnetic. If the susceptibility is positive, the material is either paramagnetic, ferromagnetic, ferrimagnetic or antiferromagnetic.

In the simplest case of a linear, isotropic and homogeneous material, the susceptibility is a dimensionless scalar. However, in more complex materials, the susceptibility may vary with the different crystallographic directions, and hence, the relation 2.4 becomes a tensorial relation with χ_{ij} a symmetric second-rank tensor, which has up to three independent components in the principal-axis coordinate system.

Inside a material, the relation 2.1 becomes

$$B = \mu H \quad (2.5)$$

where μ is the permeability of the material. It can be written as

$$\mu = \mu_0 \mu_r \quad (2.6)$$

where μ_r is the relative permeability of the material. Using relations 2.3, 2.4, 2.5 and 2.6, it is possible to link the relative permeability and the susceptibility of a material as follows:

$$\mu_r = 1 + \chi \quad (2.7)$$

Now that the different forms of magnetism have been introduced, let's focus on the main concepts that interest us for this report: ferromagnetism. The following sections will provide a comprehensive review of the characteristics of ferromagnetic materials like the hysteresis curve, the anisotropies and the ferromagnetic resonance.

2.1.3 Hysteresis curve

Ferromagnets keep a permanent magnetization with no external applied magnetic field. This spontaneous magnetism is associated with an hysteresis loop (see figure 2.1). Indeed, ferromagnetic materials show an irreversible nonlinear response of magnetization M to an imposed magnetic field H .

Ferromagnets are composed of magnetic microdomains called ferromagnetic domains. Initially, these domains are magnetized in different directions, leading to a globally zero magnetization. The domains are separated by small walls wherein the magnetization progressively rotates from the magnetization orientation of one adjacent domain to the magnetization orientation of the other adjacent domain.

When an external magnetic field is applied, the ferromagnetic domains will progressively align until the point where they are all parallel. The saturation magnetization M_s is then reached. If the external field is subsequently turned off, the magnetizations of the domains will not go back to their initial state but rather stay more or less aligned in the same direction, leading to a remanent magnetization M_r . In order to cancel out the magnetization of the material, it is necessary to decrease the field to a value $-H_c$ called coercive field. Larger hysteresis curves are associated to **hard** ferromagnets while narrower curves are associated to **soft** ferromagnets. Therefore, permanent magnets are formed with harder ferromagnets that have larger remanent magnetization.

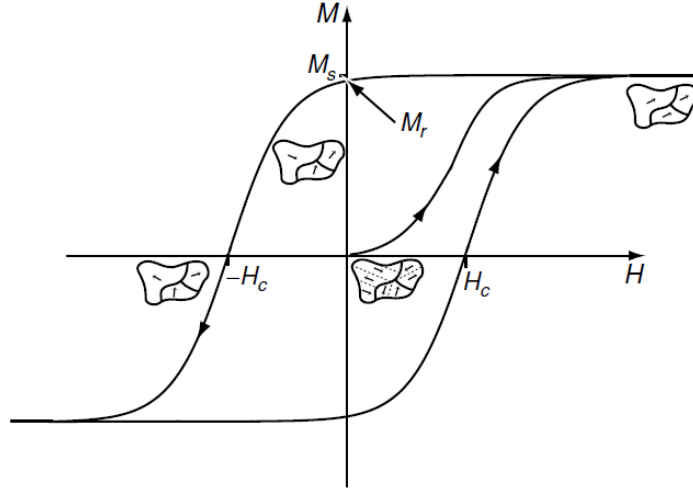


Figure 2.1: Hysteresis curve of a ferromagnetic material. H_c is the coercive field, M_r the remanent magnetization and M_s the saturation magnetization. The small schemes represent the ferromagnetic domains. At $M = 0$ and $H = 0$, the magnetizations are oriented in different directions. At large values of H , when all domains have parallel magnetizations then $M = M_s$. At $H = 0$ and $M = M_r$, the domains have magnetizations that are no longer parallel but globally oriented in the same direction. At $H = -H_c$ and $M = 0$, the domains have again completely different magnetization orientations [4].

As stated before, ferromagnetism appears only under the Curie temperature, specific for each material. Above this temperature, the thermal agitation prevents any kind of ordering of the magnetic moments. The Curie temperature of all the materials used in this work is much larger than the room temperature.

Since $M(H)$ is not a linear function in the case of ferromagnetic materials, the susceptibility is also a function of H .

2.1.4 Anisotropies

The spontaneous magnetization orientation in a ferromagnetic domain is usually constrained to lie along one or more easy axes. The energy is then minimized for a magnetization lying along these axes. The anisotropy E_a energy needed for the magnetic moment to orientate M in a direction forming an angle θ with the easy axis is given by

$$E_a = K_u \sin^2 \theta \quad (2.8)$$

Where K_u is the anisotropy constant, expressed in Jm^{-3} .

An anisotropy field H_a is usually associated to the magnetic anisotropies. It is defined as the field needed to saturate the magnetization of a uniaxial crystal in a hard magnetization direction.

A few different contributions to the anisotropy constant can be identified. The most important of them for this report are described hereafter.

Zeeman energy

The Zeeman energy describes the interaction between a magnetization M and an external applied field H leading to alignment of the magnetization with the field. For a material of volume V ,

this coupling results in an energy contribution K_z

$$K_z = -\mu_0 \int_V \vec{M} \cdot \vec{H} dV \quad (2.9)$$

Magnetostatic anisotropy

The magnetostatic anisotropy is also called shape anisotropy because it is dependent on the shape of the considered ferromagnetic materials. In the case of samples constituted by separated magnetic elements, the magnetic interactions between the elements must also be taken into account to express the magnetostatic anisotropy.

First, let's develop the influence of the shape. As mentioned before, inside a magnet, the field H has a contribution H_{dem} called demagnetizing field. This demagnetizing field tends to align with the longest dimension of a sample. In ferromagnets, H_{dem} is opposed to the orientation of the magnetization and can be linked to it through the following formula:

$$\vec{H}_{dem} = -\overline{\overline{N}} \vec{M} \quad (2.10)$$

where $\overline{\overline{N}}$ is a 3x3 symmetric tensor containing the coefficients of the demagnetizing field. In the case of an isotropic material, $\overline{\overline{N}}$ is diagonal and written as follows:

$$\overline{\overline{N}} = \begin{pmatrix} N_{xx} & 0 & 0 \\ 0 & N_{yy} & 0 \\ 0 & 0 & N_{zz} \end{pmatrix} \quad (2.11)$$

with N_{xx} , N_{yy} and N_{zz} the demagnetizing coefficients along axes x , y and z respectively and

$$N_{xx} + N_{yy} + N_{zz} = 1 \quad (2.12)$$

N_{xx} , N_{yy} and N_{zz} are well-known for some specific cases like the sphere ($N_{xx} = N_{yy} = N_{zz} = 1/3$), the infinite cylinder ($N_{xx} = N_{yy} = 1/2$ and $N_{zz} = 0$ if the long axis is along z) or a thin disk ($N_{xx} = N_{yy} = 0$ and $N_{zz} = 1$ if the face of the disk is perpendicular to z).

The magnetostatic contribution to the anisotropy K_{ms} is then written as follows

$$K_{ms} = -\frac{1}{2} \mu_0 \vec{M} \cdot \vec{H}_{dem} = \frac{1}{2} \mu_0 \vec{M} \cdot \overline{\overline{N}} \vec{M} \quad (2.13)$$

Magnetic nanowires have a very large aspect ratio (much much longer than wide) and can therefore be considered as infinite cylinders. The term K_{ms} can therefore be expressed as follows for a single nanowire with the length along the z axis:

$$\begin{aligned} K_{ms} &= \frac{1}{2} \mu_0 \begin{pmatrix} M_x \\ M_y \\ M_z \end{pmatrix} \cdot \begin{pmatrix} 1/2 & 0 & 0 \\ 0 & 1/2 & 0 \\ 0 & 0 & 0 \end{pmatrix} \begin{pmatrix} M_x \\ M_y \\ M_z \end{pmatrix} \\ &= \frac{1}{2} \mu_0 \begin{pmatrix} M_x \\ M_y \\ M_z \end{pmatrix} \cdot \begin{pmatrix} M_x/2 \\ M_y/2 \\ 0 \end{pmatrix} \\ &= \frac{1}{4} \mu_0 (M_x^2 + M_y^2) \end{aligned} \quad (2.14)$$

Since a material always tends to minimize its internal energy, magnetic nanowires will spontaneously have zero magnetization in x and y and hence a magnetization in the z direction. Single magnetic domain nanowires with a largely dominant shape anisotropy are therefore spontaneously

magnetically saturated in the z direction without needing any external applied field.

Let's now consider the case of a body composed of multiple magnetic elements. In this case, magnetic dipolar interactions have to be also considered for the determination of K_{ms} . Indeed, the magnetic charges on the surfaces of two elements interact as represented on figure 2.2. Hence, a dipolar field H_D linked to a dipolar interaction energy is induced.

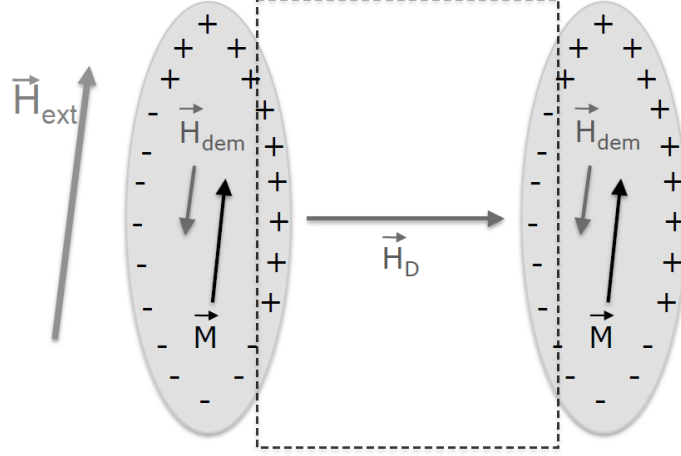


Figure 2.2: Schematic representation of two magnetic bodies subjected to a magnetic field H_{ext} . A magnetization M appears in the material as well as an opposite demagnetizing field H_{dem} . A dipolar field H_D is also induced between the bodies [6].

The dipolar interaction energy K_D between two bodies i and j with the same volume can be calculated by the formula

$$K_{D_{ij}} = -\mu_0 \vec{M}_i \cdot \vec{H}_{D_{ij}} \quad (2.15)$$

where $K_{D_{ij}}$ is the energy on the body i due to the interactions with j , $\vec{M}_i = M_s V \vec{m}_i$ is the magnetization of body i , $\vec{m}_i$ is the unit vector and $\vec{H}_{D_{ij}}$ is the dipolar field experienced by i due to the presence of j . $\vec{H}_{D_{ij}}$ can be calculated using $\vec{r}_{ij} = \vec{r}_i - \vec{r}_j$ the distance between i and j

$$\vec{H}_{D_{ij}} = \frac{1}{4\pi} \left(\frac{3 \vec{r}_{ij} (\vec{r}_{ij} \cdot \vec{M}_j)}{\|\vec{r}_{ij}\|^5} - \frac{\vec{M}_j}{\|\vec{r}_{ij}\|^3} \right) \quad (2.16)$$

For N elements, the total dipolar energy K_D is then

$$K_D = \frac{1}{2} \sum_{i=1}^N \sum_{j \neq i}^N K_{D_{ij}} \quad (2.17)$$

For arrays of magnetic nanowires like nanoporous membranes filled with nanowires, it is difficult to compute the total dipolar energy considering the numerous nanowires. A simpler method giving satisfying results is the mean field approach where the demagnetizing field of the membrane filled with nanowires is taken as the linear combination between an isolated infinite cylinder and a homogeneous infinite disk, for which the demagnetizing tensors are known [11]. These 2 cases represent the 2 extreme cases: if the packing of the nanowires (corresponding to the porosity of the membrane) tends to zero, we have only one infinitely thin nanowire, if the packing tends to 1, we have only one infinitely wide nanowire which is equivalent to a disk.

For an infinitely thin nanowire, the amplitude of the magnetostatic anisotropy field H_{ms}^{cyl} is simply

$$H_{ms}^{cyl} = \frac{1}{2}M_s \quad (2.18)$$

whereas for the disk, the amplitude of the magnetostatic anisotropy field H_{ms}^{disk} is

$$H_{ms}^{disk} = M_s \quad (2.19)$$

We now have to add these 2 contributions using the mean-field approach. The infinite cylinder should be the only term left when the porosity $p = 0$ while the disk should be the case when $p = 1$. Note also that these contributions are not in the same direction and have thus opposite signs. Hence, we can write

$$H_{ms} = H_{ms}^{cyl}(1 - p) - H_{ms}^{disk}p = \frac{1}{2}M_s(1 - p) - M_sp = \frac{1}{2}M_s - \frac{3}{2}M_sp = \frac{1}{2}M_s(1 - 3p) \quad (2.20)$$

It has been experimentally shown that this approximation was very close from the real values [11]. Note that in this equation, the term $\frac{1}{2}M_s$ can be associated to the demagnetizing field of the nanowires while the term $\frac{3}{2}M_s$ can be associated to the dipolar interaction field between the nanowires [11].

Magnetocrystalline anisotropy

Magnetocrystalline anisotropy originates from two different sources. The first contribution is a "single-ion anisotropy". The field and the crystal interact and tend to stabilize a particular orbital containing magnetic electrons. Then, the magnetic moment is aligned in a particular crystallographic direction by spin-orbit interaction. The second contribution is called "two-ion anisotropy". It is due to dipole-dipole interactions. Indeed, the lower configuration in energy is the head-to-tail configuration for two dipoles. This effect has to be summed over the entire lattice. However, this term vanishes for some lattices like cubic lattices. Some spin-orbit coupling also lead to preferred orientations of the exchange coupled pairs.

The magnetocrystalline anisotropy K_{mc} for cubic crystals (fcc, ccp) can be expressed as follows:

$$K_{mc} = K_1(\alpha_x^2\alpha_y^2 + \alpha_y^2\alpha_z^2 + \alpha_z^2\alpha_x^2) + K_2\alpha_x^2\alpha_y^2\alpha_z^2 \quad (2.21)$$

with K_1 and K_2 two anisotropy constants, α_i ($i = x, y$ or z) the directional cosines of the magnetization with respect to the [100], [010] and [001] crystallographic axes parallel to the coordinates x , y and z respectively. By replacing the angles θ and ϕ (determining the direction of the magnetization vector) into the directions cosines, we have

$$K_{mc} = \frac{1}{4}K_1(\sin^4\theta\sin^2(2\phi) + \sin^2(2\theta)) + \frac{1}{16}K_2\sin^2(\theta)\sin^2(2\theta)\sin^2(2\phi) \quad (2.22)$$

In most of the magnetic materials with cubic structure, K_1 and K_2 are at least one order of magnitude smaller than the shape anisotropy. This is for example the case for Ni and Fe that will be used in the experiments [12].

In the case of hexagonal crystals (hcp) the magnetocrystalline anisotropy is different. The easy magnetization direction is along the c-axis and the plane of the base is the hard magnetization direction. Consequently, the magnetocrystalline energy only depends on the angle θ between the orientation of the magnetization and the c-axis and some constants K_i .

$$K_{mc} = K_0 + K_1\sin^2\theta + K_2\sin^4\theta + K_3\sin^6\theta + \dots \quad (2.23)$$

In this case, K_{mc} is no longer negligible compared to the shape anisotropy. This is for example the case for Co where the orientation of the c-axis compared to the axe of nanowires can be modified using solutions for electrodeposition with different pH value. Hence, it is possible to highly vary the anisotropy field in these nanowires [13].

Magnetoelastic anisotropy

The magnetoelastic anisotropy appears for magnetostrictive materials under strain. In the case of nanowired membranes, when the membrane is cooled down, the appearing constraints generally induce an important magnetoelastic anisotropy. Indeed, since the membrane and the nanowires do not necessarily have the same thermal expansion coefficient, important constraints on the nanowires can appear.

The magnetoelastic anisotropy K_{me} is expressed as

$$K_{me} = |\sigma|\lambda \quad (2.24)$$

with σ the applied constraint and λ the magnetostriction defined as the fractional change of length $\delta l/l$.

2.1.5 Ferromagnetic resonance

The ferromagnetic resonance is associated to the precession of the magnetic moment in ferromagnets. In general, the spins of all magnetic moments are precessing around an equilibrium position with a frequency called Larmor frequency. When an oscillating magnetic field is applied, it will perturb this precession whose frequency is usually in the range of the gigahertz or tenth of gigahertz. If the frequency of a microwave signal matches the frequency of the precession of the material, a resonance phenomenon will appear and the microwaves will be absorbed. This phenomenon is interesting to characterize magnetic materials. Indeed, it allows to determine the saturation magnetization of the materials, the anisotropies, etc. From a device point of view, this phenomenon allows to filtrate some specific frequencies of microwave signals.

Larmor frequency

When an electron is subjected to a magnetic field $\vec{H}$, this field acts on the spin magnetic moment $\vec{m}$ of the electron by exerting a torque $\vec{\Gamma}$

$$\vec{\Gamma} = \mu_0 \vec{m} \times \vec{H} \quad (2.25)$$

Note that the magnetic field $\vec{H}$ can have components due to the application of an external magnetic field and due to the anisotropies.

The magnetic moment can be written as

$$\vec{m} = -\gamma \vec{l} \quad (2.26)$$

where $\vec{l}$ is the angular momentum and γ is the gyromagnetic ratio for the spin moment of an electron defined as follows:

$$\gamma = \frac{qg}{2m_e} = 175.9 [GHz/T] \quad (2.27)$$

with q the charge of the electron ($1.6 \cdot 10^{-19} [C]$), g the Landé factor worth 2 for the free electron and m_e the mass of the electron ($9.109 \cdot 10^{-31} [kg]$).

With Newton's law for the angular momentum $\vec{\Gamma} = \partial \vec{l} / \partial t$, we have:

$$\frac{\partial \vec{m}}{\partial t} = -\gamma \mu_0 \vec{m} \times \vec{H} \quad (2.28)$$

Hence, the variation of the magnetic moment with time is perpendicular to $\vec{m}$ and $\vec{H}$ leading to a precession movement (represented on figure 2.3) of angular frequency ω_L

$$\omega_L = \mu_0 \gamma H \quad (2.29)$$

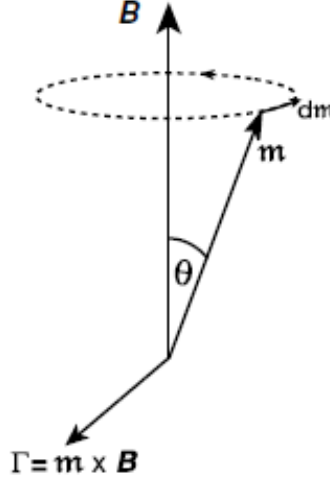


Figure 2.3: Schematic of the Larmor precession for a magnetic moment m in a magnetic field B [4].

Resonance in infinite ferromagnetic materials

For infinite ferromagnetic materials (i.e. with negligible anisotropies), we have to consider the total magnetization $\vec{M}$ which is simply defined as the sum of the magnetic moments of the material per unit volume

$$\vec{M} = \frac{1}{V} \sum \vec{m} \quad (2.30)$$

The equation for the precession of the magnetization is similar to equation 2.28 but has to account for magnetic losses due to interactions between the magnetic moments and the crystal lattice. It is then written as follows [14]

$$\frac{\partial \vec{M}}{\partial t} = -\gamma \mu_0 \vec{M} \times \vec{H} - \frac{\gamma \mu_0 \alpha}{\|\vec{M}\|} [\vec{M} \times (\vec{M} \times \vec{H})] \quad (2.31)$$

with α the damping factor usually much smaller than 1 [4]. Note also that $\|\vec{M}\| = M_s$ if the saturation is reached, which will be considered in further calculations.

It can be rewritten as

$$\gamma \mu_0 \vec{M} \times \vec{H} = -\frac{\partial \vec{M}}{\partial t} - \frac{\gamma \mu_0 \alpha}{M_s} [\vec{M} \times (\vec{M} \times \vec{H})] \quad (2.32)$$

Introducing equation 2.32 in 2.31, we have

$$\frac{\partial \vec{M}}{\partial t} = -\gamma \mu_0 \vec{M} \times \vec{H} + \frac{\alpha}{M_s} \vec{M} \times \frac{\partial \vec{M}}{\partial t} + \alpha^2 \gamma (\vec{M} \times \vec{H}) \quad (2.33)$$

because $\vec{M} \times (\vec{M} \times (\vec{M} \times \vec{H})) = M_s^2 (\vec{M} \times \vec{H})$.

Since α is small, the last term can be neglected. We have thus

$$\frac{\partial \vec{M}}{\partial t} = -\gamma \mu_0 \vec{M} \times \vec{H} + \frac{\alpha}{M_s} \vec{M} \times \frac{\partial \vec{M}}{\partial t} \quad (2.34)$$

Note that γ does not have the value given in 2.27 since we are no longer in the case of the free electron and hence the Landé factor is not strictly equal to 2.

In order to solve equation 2.34, one can use the approximation of small amplitude of precession and assume that the magnetization and the magnetic field are the sum of a steady part ($\vec{M}_0$ and $\vec{H}_0$ respectively) and an alternating part ($\vec{M}_{alt}$ and $\vec{H}_{alt}$ respectively)

$$\begin{aligned}\vec{M} &= \vec{M}_0 + \vec{M}_{alt} \\ \vec{H} &= \vec{H}_0 + \vec{H}_{alt}\end{aligned}\tag{2.35}$$

With the approximation of small amplitude of precession, we have

$$\vec{M}_{alt} \ll \vec{M}_0 \quad \vec{H}_{alt} \ll \vec{H}_0\tag{2.36}$$

We assume that $\vec{M}_0$ and $\vec{H}_0$ are oriented in the same direction. We define $\vec{M}_{alt}$ and $\vec{H}_{alt}$ as complex numbers:

$$\begin{aligned}\vec{M}_{alt} &= \vec{M}_a e^{i\omega t} \\ \vec{H}_{alt} &= \vec{H}_a e^{i\omega t}\end{aligned}\tag{2.37}$$

Using equation 2.37 in equation 2.34, we get

$$\begin{aligned}i\omega \vec{M}_a e^{i\omega t} &= -\gamma\mu_0 \left[(\vec{M}_0 + \vec{M}_a e^{i\omega t}) \times (\vec{H}_0 + \vec{H}_a e^{i\omega t}) \right] \\ &\quad + \frac{\alpha}{M_s} \left((\vec{M}_0 + \vec{M}_a e^{i\omega t}) \times (i\omega \vec{M}_a e^{i\omega t}) \right) \\ &= -\gamma\mu_0 \left[(\vec{M}_0 \times \vec{H}_0) + (\vec{M}_0 \times \vec{H}_a) e^{i\omega t} \right. \\ &\quad \left. + (\vec{M}_a \times \vec{H}_0) e^{i\omega t} + (\vec{M}_a \times \vec{H}_a) e^{2i\omega t} \right] \\ &\quad + \frac{i\omega\alpha}{M_s} (\vec{M}_0 \times \vec{M}_a) e^{i\omega t}\end{aligned}\tag{2.38}$$

Note that $(\vec{M}_0 \times \vec{H}_0) = 0$ since M_0 and H_0 are parallel. Furthermore, using the approximation described in 2.36, the term $(\vec{M}_a \times \vec{H}_a)$ can also be neglected. The equation then becomes

$$i\omega \vec{M}_a = -\gamma\mu_0 \left[(\vec{M}_0 \times \vec{H}_a) + (\vec{M}_a \times \vec{H}_0) \right] + \frac{i\omega\alpha}{M_s} (\vec{M}_0 \times \vec{M}_a)\tag{2.39}$$

Considering that $\vec{M}_0$ and $\vec{H}_0$ are oriented along the z-axis, they can be rewritten as

$$\vec{M}_0 = (0, 0, M_s)\tag{2.40}$$

and

$$\vec{H}_0 = (0, 0, H_0)\tag{2.41}$$

The two other vectors can be expressed as

$$\begin{aligned}\vec{M}_a &= (M_{ax}, M_{ay}, M_{az}) \\ \vec{H}_a &= (H_{ax}, H_{ay}, H_{az})\end{aligned}\tag{2.42}$$

Using this in equation 2.39, we obtain a system of 3 equations

$$\begin{cases} i\omega M_{ax} = \gamma\mu_0 M_s H_{ay} - \gamma\mu_0 H_0 M_{ay} - i\omega\alpha M_{ay} \\ i\omega M_{ay} = -\gamma\mu_0 M_s H_{ax} + \gamma\mu_0 H_0 M_{ax} + i\omega\alpha M_{ax} \\ i\omega M_{az} = 0 \end{cases}\tag{2.43}$$

In order to simplify the equation, the two precessions $\omega_L = \gamma\mu_0 H_0$ (Larmor precession) and $\omega_M = \gamma\mu_0 M_s$ are introduced. Solving the system gives

$$\begin{cases} M_{a_x} = \frac{(\omega_L + i\omega\alpha)\omega_M H_{a_x} + i\omega\omega_M H_{a_y}}{(\omega_L + i\omega\alpha)^2 - \omega^2} \\ M_{a_y} = \frac{-i\omega\omega_M H_{a_x} + (\omega_L + i\omega\alpha)\omega_M H_{a_y}}{(\omega_L + i\omega\alpha)^2 - \omega^2} \\ M_{a_z} = 0 \end{cases} \quad (2.44)$$

We can define χ and κ

$$\begin{aligned} \chi &= \frac{(\omega_L + i\omega\alpha)\omega_M}{(\omega_L + i\omega\alpha)^2 - \omega^2} \\ \kappa &= \frac{\omega\omega_M}{(\omega_L + i\omega\alpha)^2 - \omega^2} \end{aligned} \quad (2.45)$$

Hence, equation 2.44 becomes

$$\begin{cases} M_{a_x} = \chi H_{a_x} + i\kappa H_{a_y} \\ M_{a_y} = -i\kappa H_{a_x} + \chi H_{a_y} \\ M_{a_z} = 0 \end{cases} \quad (2.46)$$

We can now find the associated susceptibility tensor $\bar{\bar{\chi}}$ defined by

$$\vec{M} = \bar{\bar{\chi}} \vec{H} \quad (2.47)$$

Hence,

$$\bar{\bar{\chi}} = \begin{pmatrix} \chi & i\kappa & 0 \\ -i\kappa & \chi & 0 \\ 0 & 0 & 0 \end{pmatrix} \quad (2.48)$$

We can rewrite equation 2.7 for tensorial permeability and susceptibility

$$\bar{\bar{\mu}}_r = \bar{\bar{I}} + \bar{\bar{\chi}} \quad (2.49)$$

Thus, we can deduce the permeability tensor

$$\bar{\bar{\mu}}_r = \begin{pmatrix} \mu & i\kappa & 0 \\ -i\kappa & \mu & 0 \\ 0 & 0 & 1 \end{pmatrix} \quad (2.50)$$

with $\mu = 1 + \chi$.

μ and κ can be written as a sum of a real and an imaginary part

$$\begin{aligned} \mu &= \mu' - i\mu'' \\ \kappa &= \kappa' - i\kappa'' \end{aligned} \quad (2.51)$$

with

$$\begin{aligned} \mu' &= 1 + \omega_M \omega_L (\omega_L^2 - \omega^2 (1 - \alpha^2)) / D \\ \mu'' &= \omega_M \omega \alpha (\omega_L^2 + \omega^2 (1 + \alpha^2)) / D \\ \kappa' &= \omega_M \omega (\omega_L^2 - \omega^2 (1 + \alpha^2)) / D \\ \kappa'' &= 2\omega_L \omega_M \omega^2 \alpha / D \\ D &= (\omega_L^2 - \omega^2 (1 + \alpha^2))^2 + 4\omega^2 \omega_L^2 \alpha^2 \end{aligned} \quad (2.52)$$

The real and imaginary parts of κ and μ are represented on figure 2.4. A singularity appears in all curves for a frequency ω_{FMR} corresponding to

$$\omega_{FMR} = \frac{\omega_L}{1 + \alpha^2} \sim \omega_L \quad (2.53)$$

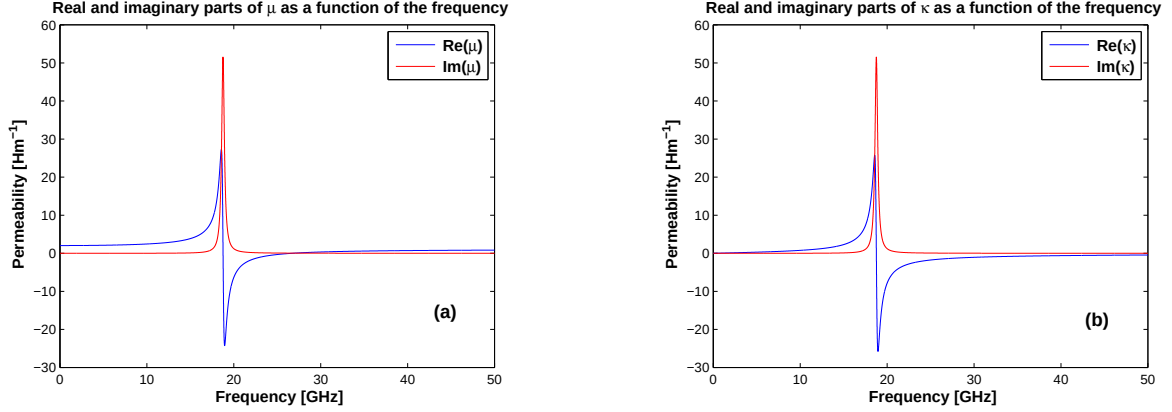


Figure 2.4: Representation of the real and imaginary part of μ (a) and κ (b) as a function of the frequency for typical values for Ni ($\gamma = 193.46 \cdot 10^9 \text{ Hz/T}$, $\alpha = 0.01$, $H = 500000 \text{ A/m}$, $M_s = 485 \cdot 10^3 \text{ A/m}$).

This frequency is called ferromagnetic resonance (FMR) frequency. It only appears in ferromagnetic materials since it needs significant magnetic moments to be non-negligible, which is only the case in ferromagnetic (and antiferromagnetic) materials. At this particular frequency, the imaginary parts of κ and μ corresponding to the magnetic losses in the medium are suddenly high. This means that an electromagnetic signal with a given frequency penetrating a ferromagnetic material with a ferromagnetic resonance at the same frequency will undergo absorption. Note also the influence of the damping as seen on figure 2.5. Larger damping constant means a broader absorption peak but also weaker absorption.

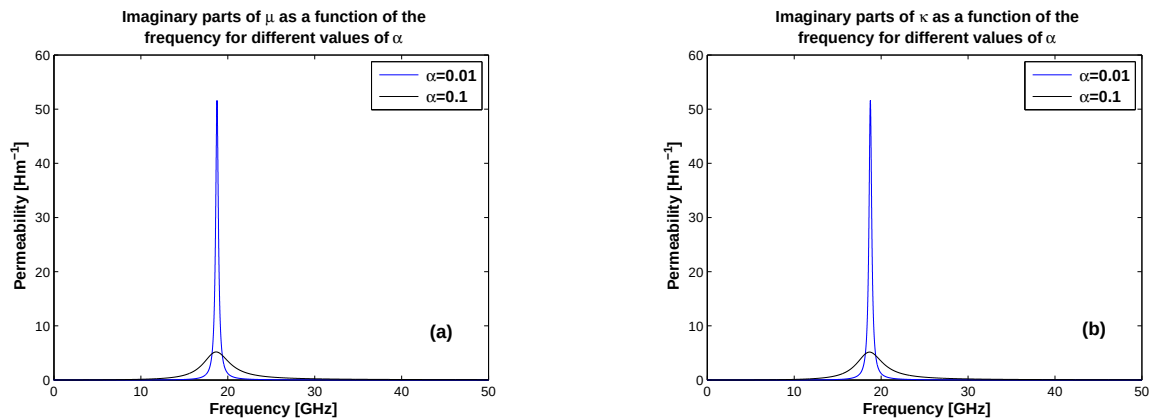


Figure 2.5: Representation of the imaginary parts of μ (a) and κ (b) as a function of the frequency for $\alpha = 0.01$ and $\alpha = 0.1$ (other values are typical for Ni: $\gamma = 193.46 \cdot 10^9 \text{ Hz/T}$, $\alpha = 0.1$, $H = 500000 \text{ A/m}$, $M_s = 485 \cdot 10^3 \text{ A/m}$).

2.1.6 Array of nanowires

Ferromagnetic Resonance

Now that the general formula for the resonant frequency has been presented, we can apply it for the case that interest us: nanowired substrates. Indeed, the previous development has been done for H an applied magnetic field. We have seen in section 2.1.4 that anisotropy fields appear in a material due to its specific properties. In the case of membranes filled with ferromagnetic nanowires, we have seen that the magnetostatic anisotropy is important. In the case of nickel and iron nanowires, the magnetocrystalline anisotropy is negligible. It is however not always the case for other materials like cobalt.

Hence, for membranes filled with ferromagnetic nanowires, the same development can be done to find the ferromagnetic resonance but we can now express separately the applied field H_{app} and the anisotropy field H_{ani} :

$$H = H_{app} + H_{ani} \quad (2.54)$$

For the case of negligible anisotropies except for the magnetostatic one, we can directly insert the magnetostatic anisotropy field given in equation 2.20 into the previous equation and use this new definition of H to find the ferromagnetic resonance frequency:

$$\omega_{FMR} = \gamma\mu_0 H = \gamma\mu_0 H_{app} + \gamma\mu_0 H_{ms} = \gamma\mu_0 H_{app} + \gamma\mu_0 \frac{1}{2} M_s (1 - 3p) = \omega_L + \frac{1}{2} \omega_M (1 - 3p) \quad (2.55)$$

If the magnetocrystalline anisotropy field is not negligible, it has to be taken into account in the definition of H_{ani} and hence it also appears in the formula of the ferromagnetic resonance frequency of an array of ferromagnetic nanowires [15].

Note in the formula that a resonance exists even if no external magnetic field is applied due to the presence of the important anisotropy field. This explains the particular interest addressed to arrays of ferromagnetic nanowires. Indeed, classic ferrites have lower anisotropy fields, leading to quasi non-existent ferromagnetic resonance in the absence of applied magnetic field. Therefore, supplementary magnets often have to be added in devices based on ferrites. Furthermore, the strong anisotropy field in arrays of ferromagnetic nanowires allows to reach even higher operating bandwidth thanks to higher saturation magnetization. This makes the use of ferromagnetic nanowired arrays particularly interesting for scaling down the devices (the membranes are small and no magnets are necessary) and increasing the operating bandwidth [1, 16].

Permeability

To compute the permeability of a nanowired substrate, one has to consider the permeability of the nanowires $\bar{\mu}_{nw}$, the permeability of the air (in the unfilled pores) $\bar{\mu}_{air}$, the permeability of the membranes $\bar{\mu}_m$, the porosity of the membrane p and the average height of the nanowires h . The global permeability $\bar{\mu}_{global}$ is then written

$$\bar{\mu}_{global} = ph\bar{\mu}_{nw} + p(1 - h)\bar{\mu}_{air} + (1 - p)\bar{\mu}_m \quad (2.56)$$

Given that the membranes used are always non-magnetic, we have $\bar{\mu}_{air} = \bar{\mu}_m = \bar{I}$ and hence

$$\begin{aligned} \bar{\mu}_{global} &= ph\bar{\mu}_{nw} + p(1 - h)\bar{I} + (1 - p)\bar{I} \\ &= ph\bar{\mu}_{nw} + (1 - ph)\bar{I} \\ &= \bar{I} + ph(\bar{\mu}_{nw} - \bar{I}) \\ &= \bar{I} + ph\bar{\chi}_{nw} \end{aligned} \quad (2.57)$$

with $\bar{\chi}_{nw}$ the susceptibility of the nanowires.

Finally, we can write the relative permeability tensor of a nanowired membrane $\bar{\bar{\mu}}_r = \bar{\bar{\mu}}_{global}$ as

$$\bar{\bar{\mu}}_r = \begin{pmatrix} \mu & i\kappa & 0 \\ -i\kappa & \mu & 0 \\ 0 & 0 & 1 \end{pmatrix} \quad (2.58)$$

with

$$\begin{aligned} \mu &= 1 + ph \frac{(\omega_{FMR} + i\omega\alpha)\omega_M}{(\omega_{FMR} + i\omega\alpha)^2 - \omega^2} \\ \kappa &= \frac{\omega\omega_M}{(\omega_{FMR} + i\omega\alpha)^2 - \omega^2} \end{aligned} \quad (2.59)$$

2.2 Electromagnetic devices for wave propagation

The following developments intend to give an overview of the propagation of electromagnetic waves in the specific structures used in this master thesis. The mathematical developments are mainly based on the book *Microwave Engineering* by D. M. Pozar [17], the book *Bases de l'ingénierie micro-ondes* by A. Vander Vorst and D. Vanhoenacker-Janvier [18] and the master thesis of Vivien Van Kerckhoven [10].

2.2.1 Rectangular Waveguide

Waveguides are structures used to guide waves like electromagnetic waves while minimizing the transmission losses. Coupled with ferromagnetic materials, it could allow to optimize the transmission of an electromagnetic signal whereas one frequency corresponding to the ferromagnetic resonance frequency would be filtered. The studied waveguide is represented on figure 2.6.

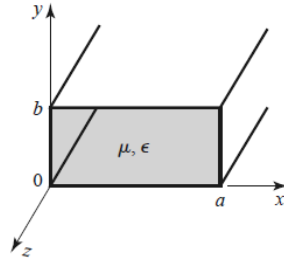


Figure 2.6: Representation of the studied rectangular waveguide of width a and height b ($a > b$) and with a medium of permeability μ and permittivity ϵ [17].

Let's first describe how electromagnetic waves act in a rectangular waveguide. For this purpose, we need to use the Maxwell equations. Considering a perfectly conducting material surrounding an homogeneous and isotropic medium and assuming a source free waveguide region, the Maxwell equations can be written as follows

$$\vec{\nabla} \cdot \vec{E} = 0 \quad (2.60)$$

$$\vec{\nabla} \cdot \vec{B} = 0 \quad (2.61)$$

$$\vec{\nabla} \times \vec{E} = -i\omega\mu\vec{H} \quad (2.62)$$

$$\vec{\nabla} \times \vec{H} = i\omega\epsilon\vec{E} \quad (2.63)$$

with ω the frequency, ϵ the permittivity, μ the permeability, $\vec{E}$ the electric field, $\vec{B}$ the magnetic field and $\vec{H}$ the H-field. Equations 2.62 and 2.63 are two equations with two unknowns $\vec{E}$ and $\vec{H}$. They can thus easily be solved for $\vec{H}$ or $\vec{E}$:

$$\vec{\nabla} \times \vec{\nabla} \times \vec{E} = -i\omega\mu\vec{\nabla} \times \vec{H} = \omega^2\mu\epsilon\vec{E} \quad (2.64)$$

Defining k

$$k = \omega\sqrt{\mu\epsilon}, \quad (2.65)$$

using the relation for any vector $\vec{A}$

$$\vec{\nabla} \times \vec{\nabla} \times \vec{A} = \vec{\nabla} (\vec{\nabla} \cdot \vec{A}) - \vec{\nabla}^2 \vec{A}, \quad (2.66)$$

and knowing that $\vec{\nabla} \cdot \vec{E} = 0$ for a source-free region, we obtain

$$(\vec{\nabla}^2 + k^2) \vec{E} = 0 \quad (2.67)$$

With a similar development, we also have

$$(\vec{\nabla}^2 + k^2) \vec{H} = 0 \quad (2.68)$$

Equations 2.67 and 2.68 are called wave equations or Helmholtz equations.

Let's now assume time-harmonic fields with an $e^{i\omega t}$ dependence and wave propagation along the z-axis. The electric and magnetic fields can then be written as

$$\begin{aligned} \vec{E}(x, y, z) &= (e_x(x, y) \vec{a}_x + e_y(x, y) \vec{a}_y + e_z(x, y) \vec{a}_z) e^{-\gamma z} \\ \vec{H}(x, y, z) &= (h_x(x, y) \vec{a}_x + h_y(x, y) \vec{a}_y + h_z(x, y) \vec{a}_z) e^{-\gamma z} \end{aligned} \quad (2.69)$$

with $\vec{a}_x$, $\vec{a}_y$ and $\vec{a}_z$ the basis vectors and γ the propagation constant that can be written as a complex number in order to take into account potential losses

$$\gamma = \alpha + i\beta \quad (2.70)$$

Maxwell equations can then be rewritten as follows

$$\begin{aligned} \frac{\partial E_z}{\partial y} + \gamma E_y &= -i\omega\mu H_x \\ -\gamma E_x - \frac{\partial E_z}{\partial x} &= -i\omega\mu H_y \\ \frac{\partial E_y}{\partial x} - \frac{\partial E_x}{\partial y} &= -i\omega\mu H_z \\ \frac{\partial H_z}{\partial y} + \gamma H_y &= i\omega\epsilon E_x \\ -\gamma H_x - \frac{\partial H_z}{\partial x} &= i\omega\epsilon E_y \\ \frac{\partial H_y}{\partial x} - \frac{\partial H_x}{\partial y} &= i\omega\epsilon E_z \end{aligned} \quad (2.71)$$

These equations can be simplified to the followings

$$\begin{aligned} H_x &= \frac{1}{k_c^2} \left(i\omega\epsilon \frac{\partial E_z}{\partial y} - \gamma \frac{\partial H_z}{\partial x} \right) \\ H_y &= \frac{-1}{k_c^2} \left(i\omega\epsilon \frac{\partial E_z}{\partial x} + \gamma \frac{\partial H_z}{\partial y} \right) \\ E_x &= \frac{-1}{k_c^2} \left(\gamma \frac{\partial E_z}{\partial x} + i\omega\mu \frac{\partial H_z}{\partial y} \right) \\ E_y &= \frac{1}{k_c^2} \left(-\gamma \frac{\partial E_z}{\partial y} + i\omega\mu \frac{\partial H_z}{\partial x} \right) \end{aligned} \quad (2.72)$$

with $k_c^2 = k^2 + \gamma^2$ the cutoff wave number.

From the Helmholtz equations 2.67 and 2.68, it is possible to express E_z and H_z as

$$\left(\frac{\partial^2}{\partial x^2} + \frac{\partial^2}{\partial y^2} + \frac{\partial^2}{\partial z^2} + k^2 \right) H_z = 0 \quad (2.73)$$

$$\left(\frac{\partial^2}{\partial x^2} + \frac{\partial^2}{\partial y^2} + \frac{\partial^2}{\partial z^2} + k^2 \right) E_z = 0 \quad (2.74)$$

Since $H_z(x, y, z) = h_z(x, y)e^{-\gamma z}$ and $E_z(x, y, z) = e_z(x, y)e^{-\gamma z}$, equations 2.73 and 2.74 can be reduced to two-dimensional wave equations

$$\left(\frac{\partial^2}{\partial x^2} + \frac{\partial^2}{\partial y^2} + k_c^2 \right) h_z = 0 \quad (2.75)$$

$$\left(\frac{\partial^2}{\partial x^2} + \frac{\partial^2}{\partial y^2} + k_c^2 \right) e_z = 0 \quad (2.76)$$

Since E_z and H_z are not coupled to transverse components E_x , E_y , H_x and H_y , it is convenient to determine solutions for the system. It is possible to study the transverse electrical modes (TE) where $E_z = 0$ and the transverse magnetic modes (TM) where $H_z = 0$. Linear combinations of these cases give the general solution. It will also be shown that no transverse electromagnetic modes (TEM) can propagate in rectangular waveguides.

TE modes

TE modes are characterized by a zero component of the longitudinal electric field $E_z = 0$. The component h_z must satisfy equation 2.75. This equation can be solved using separation of variables.

Let's assume

$$h_z(x, y) = X(x)Y(y) \quad (2.77)$$

Using this relation in equation 2.75, we obtain

$$\frac{1}{X} \frac{\partial^2 X}{\partial x^2} + \frac{1}{Y} \frac{\partial^2 Y}{\partial y^2} + k_c^2 = 0 \quad (2.78)$$

In order to satisfy this relation, each term of the equation must be equal to a constant. We define $k_c^2 = k_x^2 + k_y^2$ such that

$$\begin{aligned} \frac{\partial^2 X}{\partial x^2} + k_x^2 X &= 0 \\ \frac{\partial^2 Y}{\partial y^2} + k_y^2 Y &= 0 \end{aligned} \quad (2.79)$$

The general solution for $h_z(x, y)$ is then given by

$$h_z(x, y) = (A \cos k_x x + B \sin k_x x)(C \cos k_y y + D \sin k_y y) \quad (2.80)$$

with A , B , C and D constants. In order to determine these constants, we must apply boundary conditions on the electric field components tangential to the waveguide walls:

$$\begin{aligned} e_x(x, y) &= 0 & \text{at } y = 0, b \\ e_y(x, y) &= 0 & \text{at } x = 0, a \end{aligned} \quad (2.81)$$

with a the width of the waveguide and b the height (see figure 2.6).

To apply these boundary conditions, we must use the relations for E_x and E_y in equations 2.72. In the case of TE modes, these two equations are written

$$\begin{aligned} e_x &= \frac{-i\omega\mu}{k_c^2} \frac{\partial h_z}{\partial y} \\ e_y &= \frac{i\omega\mu}{k_c^2} \frac{\partial h_z}{\partial x} \end{aligned} \quad (2.82)$$

Reintroducing equation 2.80 in the previous equations, we have

$$\begin{aligned} e_x &= \frac{-i\omega\mu}{k_c^2} k_y (A \cos k_x x + B \sin k_x x) (-C \sin k_y y + D \cos k_y y) \\ e_y &= \frac{i\omega\mu}{k_c^2} k_x (-A \sin k_x x + B \cos k_x x) (C \cos k_y y + D \sin k_y y) \end{aligned} \quad (2.83)$$

Applying the boundary conditions in these 2 equations imposes $D = 0$, $k_y = n\pi/b$ (from first equation), $B = 0$ and $k_x = m\pi/a$ (from second equation) with $n = 1, 2, \dots$ and $m = 1, 2, \dots$. Therefore, the solution for $H_z(x, y, z)$ is

$$H_z(x, y, z) = A_{mn} \cos \frac{m\pi x}{a} \cos \frac{n\pi y}{b} e^{-\gamma z} \quad (2.84)$$

where $A_{mn} = AC$.

Using the previous result in equations 2.72 and remembering that $E_z = 0$ gives us the transverse field components of the TE_{mn} modes

$$\begin{aligned} E_x(x, y, z) &= \frac{i\omega\mu n\pi}{k_c^2 b} A_{mn} \cos \frac{m\pi x}{a} \sin \frac{n\pi y}{b} e^{-\gamma z} \\ E_y(x, y, z) &= \frac{-i\omega\mu m\pi}{k_c^2 a} A_{mn} \sin \frac{m\pi x}{a} \cos \frac{n\pi y}{b} e^{-\gamma z} \\ H_x(x, y, z) &= \frac{\gamma m\pi}{k_c^2 a} A_{mn} \sin \frac{m\pi x}{a} \cos \frac{n\pi y}{b} e^{-\gamma z} \\ H_y(x, y, z) &= \frac{\gamma n\pi}{k_c^2 b} A_{mn} \cos \frac{m\pi x}{a} \sin \frac{n\pi y}{b} e^{-\gamma z} \end{aligned} \quad (2.85)$$

The value of k_c^2 can also be extracted:

$$k_c^2 = \left(\frac{m\pi}{a}\right)^2 + \left(\frac{n\pi}{b}\right)^2 \quad (2.86)$$

And hence, the propagation constant γ can be rewritten

$$\gamma = \alpha + i\beta = \sqrt{k_c^2 - k^2} = \sqrt{\left(\frac{m\pi}{a}\right)^2 + \left(\frac{n\pi}{b}\right)^2 - \omega^2\mu\epsilon} \quad (2.87)$$

It is only worth zero for

$$\omega = \omega_c = \frac{1}{\sqrt{\mu\epsilon}} \sqrt{\left(\frac{m\pi}{a}\right)^2 + \left(\frac{n\pi}{b}\right)^2} = 2\pi f_{c_{mn}} \quad (2.88)$$

where $f_{c_{mn}}$ is called cutoff frequency. Each mode of propagation has a cutoff frequency. The mode with the lowest cutoff frequency is called the dominant mode. With the width of the waveguide a smaller than the height b , the dominant mode is the mode TE₁₀ with a cutoff frequency $f_{c_{10}}$

$$f_{c_{10}} = \frac{1}{2a\sqrt{\mu\epsilon}} \quad (2.89)$$

If we assume that the medium is lossless i.e. ϵ and μ are real, the propagation constant is real for $\omega < \omega_c$. We then have

$$\gamma = \alpha = \sqrt{k_c^2 - \omega^2 \mu \epsilon} \quad (2.90)$$

which means that under the cutoff frequency, all field components decay exponentially due to the term $e^{-\gamma z} = e^{-\alpha z}$. These modes are called evanescent modes.

For $\omega > \omega_c$, γ is purely imaginary since the content of the square root is negative. We then have

$$\gamma = i\beta = \sqrt{\omega^2 \mu \epsilon - k_c^2} \quad (2.91)$$

In this case, the modes will propagate without attenuation. If more than one mode propagates in the waveguide, it is said to be overmoded and interference occurs.

If μ or ϵ are not purely real numbers, the situation is more complex. There is still a cutoff of the propagation of the field components but it is much less sharp than for a lossless medium.

TM modes

TM modes are characterized by a zero component of the longitudinal magnetic field $H_z = 0$. The development is very similar to the one for TE modes. Hence, it will be kept shorter in this section.

E_z must satisfy the relation 2.76. Using the separation of variables in the same way we did for TE modes, we get the general solution for $e_z(x, y)$

$$e_z(x, y) = (A \cos k_x x + B \sin k_x x)(C \cos k_y y + D \sin k_y y) \quad (2.92)$$

The boundary conditions we have to apply are

$$\begin{aligned} e_z(x, y) &= 0 & \text{at } x = 0, a \\ e_z(x, y) &= 0 & \text{at } y = 0, b \end{aligned} \quad (2.93)$$

These conditions impose $A = 0$, $C = 0$, $k_x = m\pi/a$ and $k_y = n\pi/b$ with $n = 1, 2, \dots$ and $m = 1, 2, \dots$. The solution for $E_z(x, y, z)$ is then

$$E_z(x, y, z) = B_{mn} \sin \frac{m\pi x}{a} \sin \frac{n\pi y}{b} e^{-\gamma z} \quad (2.94)$$

where $B_{mn} = CD$.

Using the equations 2.72 and remembering that $H_z = 0$ gives us the transverse field components of the TM_{mn} modes:

$$\begin{aligned} E_x(x, y, z) &= \frac{-\gamma m \pi}{k_c^2 a} B_{mn} \cos \frac{m\pi x}{a} \sin \frac{n\pi y}{b} e^{-\gamma z} \\ E_y(x, y, z) &= \frac{-\gamma n \pi}{k_c^2 b} B_{mn} \sin \frac{m\pi x}{a} \cos \frac{n\pi y}{b} e^{-\gamma z} \\ H_x(x, y, z) &= \frac{i\omega \epsilon n \pi}{k_c^2 b} B_{mn} \sin \frac{m\pi x}{a} \cos \frac{n\pi y}{b} e^{-\gamma z} \\ H_y(x, y, z) &= \frac{-i\omega \epsilon m \pi}{k_c^2 a} B_{mn} \cos \frac{m\pi x}{a} \sin \frac{n\pi y}{b} e^{-\gamma z} \end{aligned} \quad (2.95)$$

The propagation constant is, just like for TE modes,

$$\gamma = (\alpha + i\beta)^2 = \sqrt{k_c^2 - k^2} = \sqrt{\left(\frac{m\pi}{a}\right)^2 + \left(\frac{n\pi}{b}\right)^2 - \omega^2\mu\epsilon} \quad (2.96)$$

The cutoff frequencies for TM_{mn} modes are hence the same as for TE_{mn} modes. Note however that $\vec{E}$ and $\vec{H}$ are zero if m or n is zero. Hence, there are no TM_{m0} and TM_{0n} modes and the first mode propagation is the mode TM_{11} with a cutoff frequency

$$f_{c11} = \frac{1}{2\pi\sqrt{\mu\epsilon}} \sqrt{\left(\frac{\pi}{a}\right)^2 + \left(\frac{\pi}{b}\right)^2} \quad (2.97)$$

which is larger than the cutoff frequency of the mode TE_{10} . Hence, dimensions of waveguides and operating frequencies are often chosen such that only the mode TE_{10} propagates. The cutoff frequency of the mode TM_{11} is even larger than the cutoff frequency of mode TE_{20} if $a^2/b^2 > 3$. Hence, for rectangular waveguides with large width on height ratio, the operating bandwidth of interest is between the cutoff frequency of the mode TE_{10} and the cutoff frequency of the mode TE_{20} . For higher frequencies, the waveguide is overmoded and a lot of perturbations appear.

TEM modes

TEM modes are characterized by a zero component of the electric field E_z and of the magnetic field H_z . We can see from 2.72 that all components of the electric field and the magnetic field are zero unless $k_c^2 = 0$ which is the same as $k^2 = -\gamma^2$. Hence, there is no cutoff frequency for TEM modes. From equations 2.67 and 2.68, we can deduce the Helmholtz equations for E_x , E_y , H_x and H_y . Let's first make the development for the components of the electric field.

We have for E_x

$$(\vec{\nabla}^2 + k^2) E_x = 0 \quad (2.98)$$

Since e_x has no z components (and hence E_x has its only z dependence in the term $e^{-\gamma z}$), we have $\partial^2 E_x / \partial z^2 = \gamma^2 E_x = -k^2 E_x$. Hence, it simplifies as follows

$$\left(\frac{\partial^2}{\partial x^2} + \frac{\partial^2}{\partial y^2} \right) E_x = 0 \quad (2.99)$$

Similarly, we have for E_y

$$\left(\frac{\partial^2}{\partial x^2} + \frac{\partial^2}{\partial y^2} \right) E_y = 0 \quad (2.100)$$

We can even impose, from 2.60 that $\vec{\nabla} E_x(x, y) = 0$ and $\vec{\nabla} E_y(x, y) = 0$.

Furthermore, from 2.62, knowing that $E_z = H_z = 0$ and that E_x and E_y are independent of z , we can deduce

$$\frac{\partial E_y}{\partial x} = \frac{\partial E_x}{\partial y} \quad (2.101)$$

Taking these equations together ensures that E_x and E_y are constants (independent of x , y and z).

Applying the boundary conditions 2.81 imposes $E_x = E_y = 0$. Using this result in 2.71 also induces $H_x = H_y = 0$. Hence, we conclude that no propagation of TEM waves are possible in a rectangular waveguide. It can be shown that TEM waves only exist when two or more conductors are present.

Impedance

The impedance of a waveguide is particularly complex to describe. Different definitions exist, giving different values of impedance. In order to have good transmissions of electromagnetic waves coming for example from transmission lines to the waveguide, it is however important to ensure impedance adaptation.

The intrinsic impedance η of a medium of permeability μ and permittivity ϵ is given by the relation

$$\eta = \sqrt{\frac{\mu}{\epsilon}} \quad (2.102)$$

The wave impedance is characteristic for each wave. TE waves, TEM waves or TM waves have different wave impedance. The global definition for the wave impedance Z_w of a rectangular waveguide is

$$Z_w = \frac{E_x}{H_y} = \frac{-E_y}{H_x} \quad (2.103)$$

In the case of TE modes described above, for frequencies larger than the cutoff frequency (γ purely imaginary), this gives

$$Z_w = \frac{i\omega\mu}{\gamma} = \frac{k\eta}{\beta} \quad (2.104)$$

This impedance is independent of the height of the waveguide. This could lead to think that b is not important in the impedance of a waveguide and hence its adaptation with for example transmission lines. However, experiments show that changes in the height of rectangular waveguides induce mismatch. This happens because the given definition of impedance doesn't account for the longitudinal components of the magnetic or electric fields which are never both zero in modes propagating in a rectangular waveguide as we have seen before. The power carried by the wave changes with the height of the waveguide.

Other definitions of the impedance based on the potential V , the current I and the power P in the waveguide exist. We can find V , I and P for the mode TE_{10} . The voltages between the opposite walls can be found integrating the electric field on the distance between these walls in the waveguide. The surface current on the different walls can be calculated using the cross product of the magnetic field at the position of the wall ($x = 0$ or $x = a$ or $y = 0$ or $y = b$) and the normal to the wall. Finally, the power can be computed with the following formula

$$P = \frac{1}{2} \int_0^a \int_0^b \vec{E} \times \vec{H}^* \cdot \vec{z} dy dx \quad (2.105)$$

However, values computed for I , V and P in the case of the rectangular waveguides don't observe the relation $P = (1/2)VI^*$ where I^* is the complex conjugate of the current [18]. Hence multiple definitions of the characteristic impedance of the waveguide exist:

$$Z_{VI} = \frac{V}{I} \quad Z_{PI} = \frac{2P}{|I|^2} \quad Z_{PV} = \frac{|V|^2}{2P} \quad (2.106)$$

Since the impedance of the waveguide is not clearly defined, only different approximations of the impedance can be done and a real match impedance is hard if not impossible to obtain. Hence, some structures specific to the waveguides used will have to be considered.

Substrate Integrated Waveguides

This section is mainly based on the reviews by M. Bozzi, A. Georgiadis, K. Wu [19] and from A. Dahiya [20].

Waveguides are of primary importance when it comes to power transmission between electronic components. The constraints to improve waveguides are the same as those for other electronic components: higher performances, higher densities, lower costs and more reliable techniques. Substrate integrated waveguides or SIW are an interesting alternative to classical waveguides. They are particularly interesting to at least reduce the size of commonly used waveguides and maybe even reduce the cost while keeping their performances.

SIW are composed of two rows of conducting cylinders or slots embedded in a dielectric substrate that connects two parallel metal plates, as represented on figure 2.7.

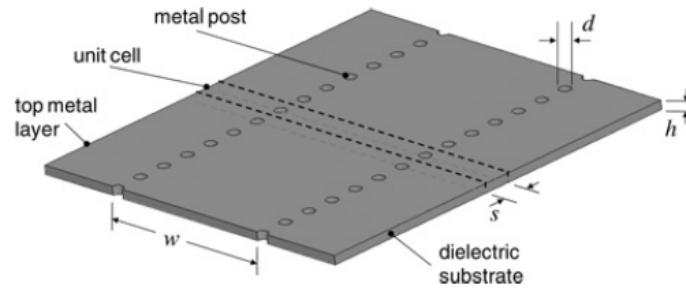


Figure 2.7: SIW of width w composed of two parallel metallic layers separated by a dielectric substrate and two rows of metal posts of diameter d and unit cell of size s . The height of the substrate is h [19].

The propagation in substrate integrated waveguides is similar to the one described for rectangular waveguides except that the TM modes do not propagate correctly and tend to leak because of the gaps between the metal vias. Indeed, because of the gaps, no surface current can propagate on the walls and hence the magnetic field can leak through the gaps [21]. However, as discussed before, the dominant mode in waveguides is mode TE_{10} and the dimensions are often chosen in order to let this mode propagate. Furthermore, it was shown that if $a^2/b^2 > 3$, which is obviously the case for thin substrates, the second mode that will propagate and disturb the propagation of the mode TE_{10} is mode TE_{20} . Hence, the operating bandwidth of SIW is, for most applications, similar to the ones for rectangular waveguides.

Other advantages of rectangular waveguides like high quality factor, shielding effect and high power-handling capacity can also be found for SIW. One possible source of performance reduction is the losses. Three sources of losses are present in SIW: dielectric, conductor and radiation losses. The two first sources also appear in rectangular waveguides while the third is specific to SIW. The dielectric losses are inherent to the material filling the waveguide and are thus the same for SIW and rectangular waveguides. Conductor losses are almost inversely proportional to the height of the substrate. It is an important problem since one of the main advantages of SIW is the size reduction compared to traditional waveguides. Finally, the radiation losses are due to leakage between the metal vias. They have been shown to be small if $s/d < 2.5$ [19].

A problem presented before for rectangular waveguides that also applies for SIW is the problem of correct impedance definition that induces difficulties to match the waveguide with another

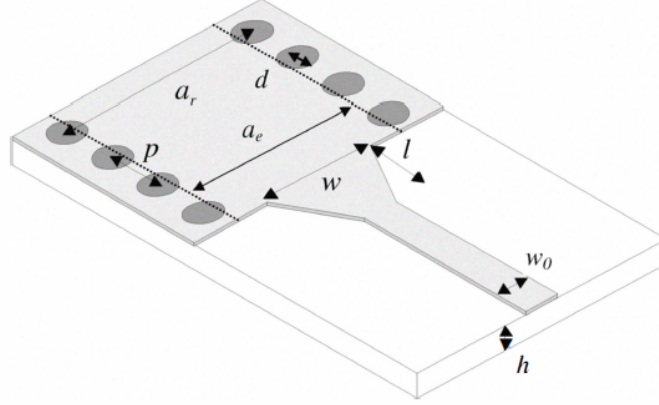


Figure 2.8: SIW matched to a microstrip line of width w_0 through a taper of length l and width w_0 on the side of the microstrip and w on the side of the waveguide [22].

structure propagating electromagnetic waves. It was shown that there are no known formula that precisely describe the impedance of rectangular waveguides. However, in the frame of this master thesis, it will be needed to correctly match a microstrip line and a SIW. Two specific structures can be used to ensure impedance match: the quarter-wave transformer [17] and a microstrip taper presented by D. Deslandes [22].

The quarter-wave transformer is a simple and useful circuit for impedance matching. It simply consists in a transmission line linking the components of the circuit (in this case the microstrip line and the waveguide) with a length corresponding to $1/4$ of the wavelength associated to the frequency at which we wish to have a match. We assume that we know the real load resistance R_L (of the waveguide) and the real feedline characteristic impedance Z_0 (of the microstrip line, usually equal to 50Ω). The 2 components are connected by a lossless piece of transmission line of (unknown) characteristic impedance Z_1 and length $\lambda/4$. We want to match the load to the microstrip line of impedance Z_0 . The input impedance Z_{in} is

$$Z_{in} = Z_1 \frac{R_L + iZ_1 \tan \beta l}{Z_1 + iR_L \tan \beta l} \quad (2.107)$$

with $\beta l = (2\pi/\lambda)(\lambda/4) = \pi/2$. Dividing the numerator and the denominator by $\tan \beta l$ and taking the result for $\beta l \rightarrow \pi/2$, we have

$$Z_{in} = \frac{Z_1^2}{R_L} \quad (2.108)$$

Since we want to match it with a microstrip line of characteristic impedance Z_0 , it gives as result

$$Z_1 = \sqrt{R_L Z_0} \quad (2.109)$$

We don't know the value of R_L for waveguides but it is possible to converge that value by simulation as it will be shown later on. Note also that the match of impedance is only perfect for one frequency and hence mismatches appear for other frequencies.

D. Deslandes introduced a taper to match the impedance of a microstrip line and a SIW, as represented on figure 2.8.

Some formulas are proposed in the article from D. Deslandes, however we did not find interesting to introduce them here since they led to contradictions with values of w and l found from simulation. Furthermore, examples described in the article did not seem to use values indeed deduced from these formulas.

2.2.2 Microstrip lines

This section is entirely based on the description made in the book *Microwave Engineering* by D. M. Pozar [17].

Microstrip lines are simple and popular planar transmission lines. They can be easily fabricated by photolithographic or metal deposition process. They consist in a conductive line of width W deposited on a thin grounded dielectric substrate of thickness d and relative permittivity ϵ_r . A microstrip line is represented on figure 2.9.

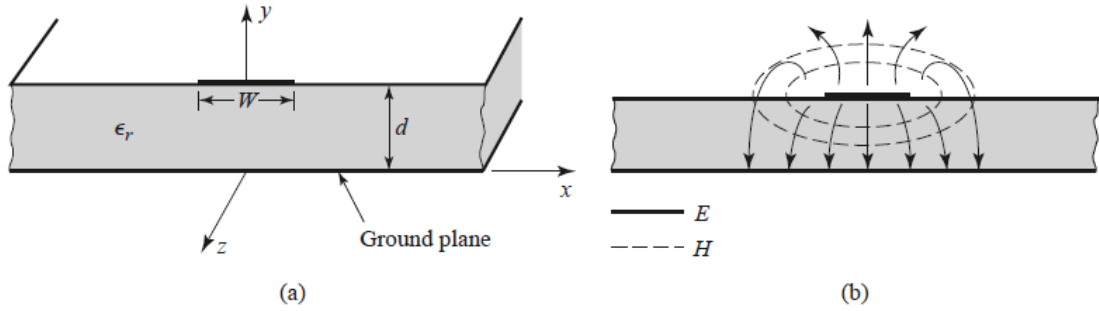


Figure 2.9: Representation of (a) the geometry of a microstrip line of width W , thickness d and relative permittivity ϵ_r and (b) the electric and magnetic field lines [17].

The analysis of microstrip lines is particularly hard to perform due to the presence of field lines in the dielectric substrate below the conductive line and in the air above the conductive line. A description or approximation of the behavior of microstrip lines includes the introduction of the effective dielectric constant ϵ_e to account for this. Hence, ϵ_e satisfies the relation

$$1 < \epsilon_e < \epsilon_r \quad (2.110)$$

A good approximation of the effective dielectric constant and the impedance of the microstrip line is given by the following phenomenological formulas:

$$\epsilon_e = \frac{\epsilon_r + 1}{2} + \frac{\epsilon_r - 1}{2} \frac{1}{1 + 12d/W} \quad (2.111)$$

$$Z_0 = \begin{cases} \frac{60}{\sqrt{\epsilon_e}} \ln\left(\frac{8d}{W} + \frac{W}{4d}\right) & \text{for } W/d \leq 1 \\ \frac{120\pi}{\sqrt{\epsilon_e} [W/d + 1.393 + 0.667 \ln(W/d + 1.444)]} & \text{for } W/d \geq 1 \end{cases} \quad (2.112)$$

Chapter 3

Experimental methods and instruments

In this chapter, the experimental methods and instruments used in the frame of this master thesis will be described. In particular, the processes for the preparation of the membrane and the growing of the nanowires will be described, along with the instruments used for the fabrication of a complete device and the characterization techniques.

3.1 Aluminum anodizing process

The anodizing of aluminum in acid solutions produces self-ordered nanoporous alumina (aluminum oxide). The pores appear with a high density in a self organized hexagonal structure during the pore growth. Hence, it can be used as template for nanosize structures [23–27]. The bottom of typical anodic alumina is represented on figure 3.1.

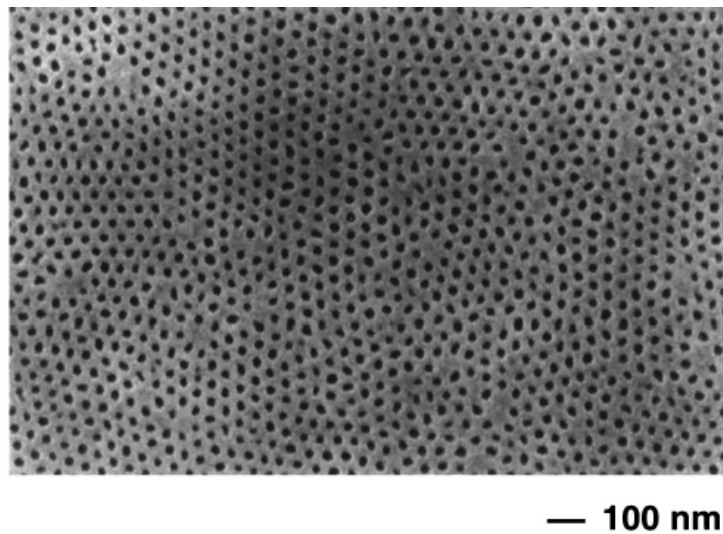


Figure 3.1: SEM bottom view of a porous alumina layer prepared in sulfuric acid (20wt.%, H_2SO_4 , 18.7V, 1°C) [24].

The anodization process consists in submerging aluminum in an acid bath (oxalic, sulfuric, and phosphoric acid solutions [23]) and applying an electrical potential using the aluminum as the anode and a cathode in stainless steel placed on top of the membrane. The surface of the aluminum then oxidizes due to the migration of oxygen-containing ions (OH^- , O^{2-}) from the solution to the aluminum. Thereafter, positive aluminum ions (Al^{3+}) move to the acid/alumina interface while oxygen-containing ions move to the alumina/aluminum interface. This process leads to the formation of nanosized pores that grow vertically from the surface of the aluminum and are surrounded by oxidized aluminum (represented on figure 3.2). Note that the atomic density is about two times smaller in alumina than in aluminum leading to an expansion of

the material when anodized, accentuated by the formation of the pores. However, the final volume of the anodized alumina is less than two times larger than the initial aluminum volume since aluminum ions also leave the material during the anodization process. When the required height of pores is obtained, the remaining aluminum can be removed using a saturated HgCl_2 solution. An alumina layer known as barrier layer always remains around the pores, preventing the complete opening of these at the bottom of the membrane. This can be solved by chemical etching using a phosphoric acid solution. The duration of this etching must however be strictly controlled since it also induces pores enlargement [23].

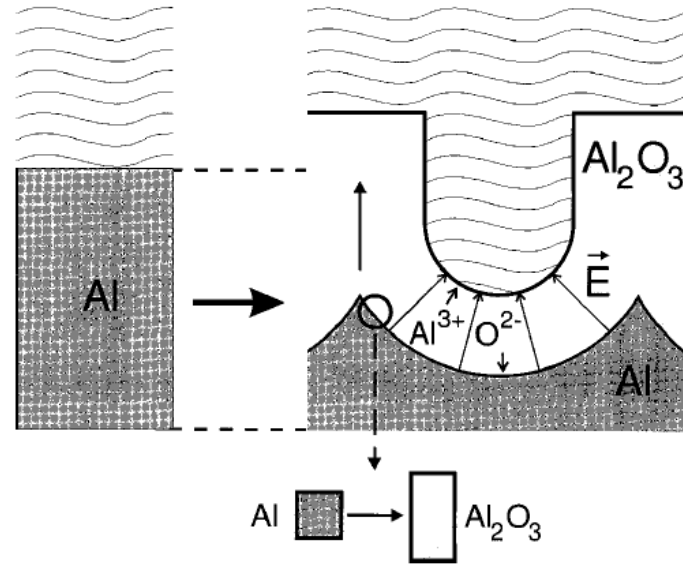


Figure 3.2: Representation of the pore growth during the anodization process on an aluminum membrane. Oxygen ions are moving toward the aluminum while aluminum ions are moving toward the solution [24].

While the mechanism of self-ordering of the pores in hexagonal structure arrays is not fully understood, Jessensky et al. proposed an explanation based on the mechanical stresses appearing during the anodization process [24]. Indeed, since the atomic density of alumina is twice lower as in aluminum and since alumina appears on the whole surface of aluminum during the process, the latter undergoes compressive stress. This compressive stress is assumed to be the origin of the repulsive force between neighboring pores leading to the organized structure. It was observed that a regular pore formation only occurs for a thickness ratio of alumina to consumed Al close to 1.4 (ratio of the thickness after and before anodization) [23]. The specific voltages, temperatures and electrolyte concentrations are supposed to vary the mechanical stresses and hence only specific values lead to an efficient anodization. Note also that acid solutions are used as electrolytes because it was shown that basic and neutral solutions didn't lead to pore formations [27]. The acid acts here as a catalyst for the pore growth.

The self-ordering of the nanopores doesn't occur perfectly directly at the beginning of the process. It takes some time for the pores to perfectly align in hexagonal structure arrays and hence, the top surface of the resulting alumina doesn't show this well-organized structure. A way to obtain perfect order at the surface was proposed by Masuda et al. in 1996 and is now called double anodization or two-step anodization [25]. It consists in a first anodization step stopped when the pores begin to align. Then, the top porous alumina is removed using a mixture of phosphoric and chromic acid. The remaining aluminum is slightly etched at the surface due to the growth of

the pores in the removed part. A second anodization step is subsequently performed. The pores naturally initiate their growth on the organized etches. This leads to anodic porous alumina with ordered pores in hexagonal structure on the upper and lower sides.

The nanoporous alumina membranes will be used here rather than the other well-known kind of nanoporous template: the track-etched polycarbonate membranes [28]. In these membranes, the nanopores are produced thanks to a bombardment of accelerated heavy ions (typically in a cyclotron) and the subsequent etching of the tracks left by the ions. While it also makes possible the growth of nanowires, it has some disadvantages compared to the anodized alumina membranes particularly for microwave applications that are central in this work [29]. Indeed, polymer membranes are not compatible with cleanroom processes and can hence not be introduced into the fabrication of a complete device contrarily to alumina membranes. Furthermore, polymer membranes show much more dielectric losses leading to larger insertion losses than alumina membranes. The thickness of metal deposited on polycarbonate is also restrained due to strain mismatch between the polymer and the metal. Thus, only metallic layers thinner than a few hundred of nanometers can be deposited for the ground plane or microstrip lines, which leads to important conduction losses. Finally, alumina membranes show large isolation and their regular ordering makes the description of dipolar interaction between magnetic nanowires much easier. All together, it clearly shows that anodized alumina membranes are much more adapted for microwave applications than track-etched polycarbonate membranes.

During this work, three kinds of nanoporous alumina membranes were fabricated or used: membranes fabricated by Smart Membranes (that will be referred to as SM), membranes fabricated in the labs of UCL and membranes fabricated by Synkera (that will be referred to as Sy).

- SM are industrially produced double anodized alumina membranes. They have a porosity of 10%, pores of $40nm$ diameter and a thickness of $100\mu m$. They are sold in sheets which allows to cut them as we want and hence obtain various sizes. However, due to the low mechanical resistance, they have to be cut using a picosecond laser (described here below) for reasonable precision. SM are the main type of nanoporous membranes used for this work.
- Anodized alumina membranes were fabricated in UCL in order to replace industrial ones. However, the resulting products weren't easily usable mainly due to very low mechanical resistance. Hence, they weren't fully characterized but the desired height was $50\mu m$ and the pore density around 30% with pores of diameters between 40 and $50nm$. The full explanation of the first experiments on these membranes and the reason they weren't used in this work are presented in appendix A.
- Sy are industrially produced double anodized alumina membranes. They have a height of $100\mu m$, a pore density of 12% and $35nm$ of pore diameter. They were mostly used to replace SM when the laser was unavailable. Their advantage is their higher mechanical resistance. Their main disadvantage is the unique size.

3.2 Electrochemical deposition

It is possible to grow nanowires in anodized alumina membranes (or track-etched polycarbonate membranes) using electrochemical depositions with a three-electrodes configuration. It consists in submerging the membrane in a solution of conductive materials and applying an electric field in order to induce an oriented diffusion of charged particles. The two electrodes used for the process are a metallic bar plunged in the solution acting as the anode and a metallic layer at

the bottom of the membrane acting as the cathode. Positive metallic ions hence diffuse to the cathode and are reduced on it, leading to a growth of the nanowires. Note that once ions begin to reduce on the bottom metallic layer, the deposited metallic particles act as the cathode instead of the metallic layer. When the pores are completely filled, the nanowires overfill the membrane and create a metallic layer by joining each other. The setup used for electrochemical depositions is represented on figure 3.3.

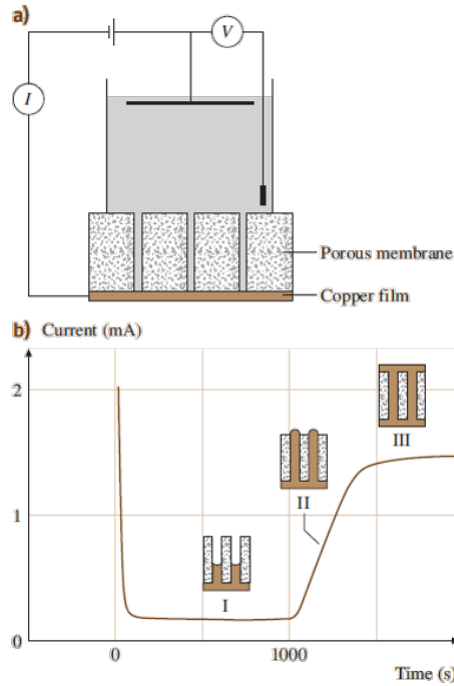


Figure 3.3: a) Representation of an electrochemical deposition cell with V the applied voltage, I the induced current and the solution in grey. b) Typical curve of an electrochemical deposition showing the deposition current as a function of the time. Three important steps can be observed: the first plateau corresponding to the filling of the pores (1D growth), the sudden increase in current due to the overfilling of the pores (leading to higher deposition surface and 3D growth) and the second plateau corresponding to a complete covering of the top of the membrane with metal [30].

The advantages of this technique are its simplicity and the possibility to have a control of the height of the nanowires through the analysis of the deposition current curve [30].

For the electrodepositions done in this master thesis, the anode was a small platinum bar while the cathode was usually a layer of $1.5\mu\text{m}$ of copper. The potential was applied between the electrodes by a potentiostat using as reference an electrode immersed in a solution containing AgCl . The potential can be extracted from the measurement of the Cl^- concentration produced by the reduction-oxidation reaction of AgCl .

Specific electric potentials are needed to reduce specific materials. Indeed the standard reduction potentials give the voltage under which the ions in solution will undergo reduction and will deposit on the cathode. The standard reduction potential of the reference which is equal to 0.22V must be subtracted to these values since the values available in the literature are computed for hydrogen reference electrodes. For example, the standard reduction equation of Ni is



and its standard reduction potential is $-0.257V$ [31]. This means that the actual voltage that has to be applied must be under $-0.257 - 0.22 = -0.477V$. Under this value, the smaller the potential, the quicker the deposition will happen. However, the quality and homogeneity of the deposition decrease if the absolute value of the potential is increased.

Note also that the deposition is not identical on all cathodes. For example, tests of Ni electrodeposition directly on aluminum and copper films (without nanoporous membrane on them) were performed in the labs. The deposition curves are shown on figure 3.4 for a total charge deposited of $4C$.

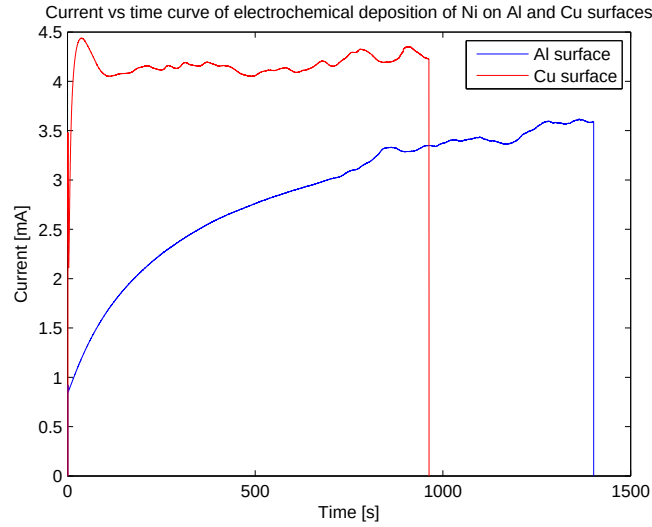


Figure 3.4: Electrochemical deposition of a charge of $4C$ of nickel on aluminum and on copper films

It is clear that the deposition on copper is quicker. We can also observe that the curve of copper deposition is directly flat. For aluminum, the current increases gradually which tends to show that the deposition becomes faster when the cathode becomes the nickel already deposited rather than the aluminum surface. This can be explained by differences in conductivity, surface rugosity or surface interactions with the deposited material on aluminum and copper.

3.3 Electron beam evaporation

Electron beam evaporation is a physical vapor deposition technique widely used to deposit thin films of materials. It is based on the use of an electron source (typically a heated tungsten filament) that produces an electron beam. This electron beam is accelerated and deflected by magnets to strike the evaporant (the material one wants to deposit) contained in a crucible. This happens in high vacuum (typically smaller than $10^{-4}mbar$) such that the mean free path of the electrons (the average distance they can travel before colliding with another particle) is longer than the distance between the electron beam source and the evaporant. When the electron beam hits the evaporant, its kinetic energy is transformed into heat via the impact. Hence the evaporant evaporates, the particles reach the substrate upwards and coat it. The height of the coating layer is measured using a crystal type film [32–34]. A scheme of the working principle is shown on figure 3.5.

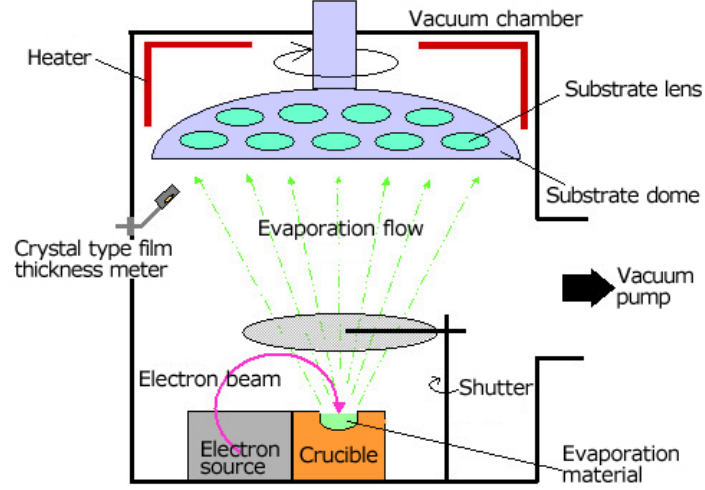


Figure 3.5: Scheme of an e-beam evaporation chamber. The electron source produces an electron beam striking the evaporation material in a crucible. The shutter opens and the evaporation flow propagates to the substrate. Vacuum is done via the vacuum pump and the height of material deposited on the substrate is measured via a crystal type film thickness meter [34].

The advantages of the electron beam evaporation technique are its high efficiency for the heating of the evaporant, its high deposition rate, and the precise control on the thickness of the coating [34]. It was used to deposit the cathodes and the microstrip lines which were typically composed of 5nm of Cr, $1.5\mu\text{m}$ of Cu and 100nm of Au. The chromium layer had to be deposited first due to the poor adhesion of copper on the membranes. Indeed, it acts as an adhesive between two layers deposited by electron beam evaporation (described hereafter) [35]. The gold layer was deposited at the end to prevent the oxidation and improve the contacts.

3.4 Scanning Electron Microscope

The scanning electron microscope (SEM) uses an electron beam produced by a heated filament to scan the surface of a sample. The beam of electrons is focused towards the surface of the sample thanks to magnetic coils. When a material is hit by electrons, it produces secondary electrons directly expelled due to the too large number of electrons in the materials. It also emits backscattered electrons for the same reason but these come from deeper in the material and undergo scattering before they are emitted. The bombardment of atoms can also cause destabilization of their electronic orbitals leading to the emission of so-called Auger electrons or x-rays. The different emissions induced by an electron bombardment are represented on figure 3.6.

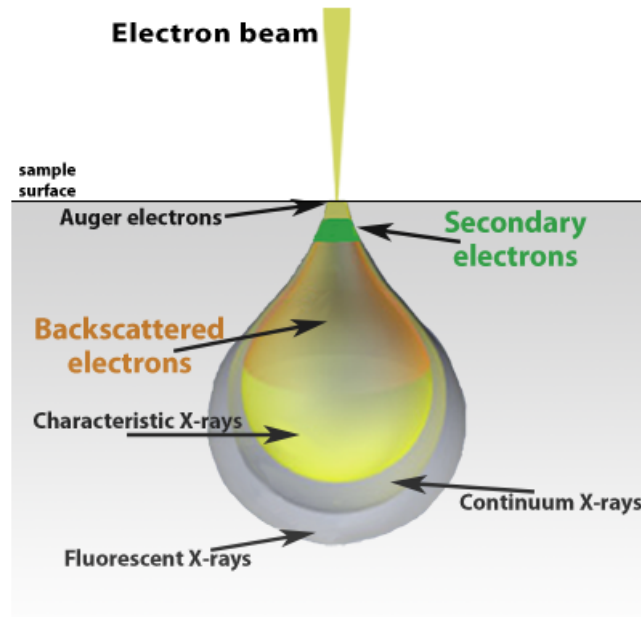


Figure 3.6: Interaction pear representing the different emissions induced by the bombardment of a material with an electron beam. The Auger electrons and secondary electrons are originary from the surface of the sample while backscattered electrons and x-rays are coming from deeper in the material (up to a few microns) [36].

The intensity of the secondary electrons reflects the topology of the surface of the sample and it is used to create an image of the surface. The SEM allows to observe surfaces with a resolution smaller than a nanometer, which is far superior to the one reached by optical microscopes. Backscattered electrons can be used to give information on the topography and the chemistry of the sample. However, in order to get an analysis of the atomic composition, the x-rays are usually rather used. The analysis performed is then called Energy-dispersive X-ray analysis (EDX) and allows to get a semi-quantitative spectrum. Analyzing this spectrum gives the elements composing the surface of the sample as well as the relative quantities of the different elements.

3.5 Picosecond Laser

Picosecond Lasers utilize very short pulses laser lasting typically from 8 to 12 picoseconds applied with a default frequency of $200kHz$. The highly energetic pulses are able to sublime a material while preventing the formation of a heat-affected zone. Hence, they can be used to drill a surface, etch a sample or precisely cut a material up to $0.5mm$ thick without or with little thermal damage [37].

The wavelength of the used laser beam is $355nm$ and the typical internal power is around 3 or 4 Watt. A few of the parameters can be tuned to match the applications. The applied power can be decreased to a percentage of the internal power and the default frequency can be divided by an integer factor. The number of passes of the laser on a pattern can also be determined along with the displacement speed of the laser beam, the vertical focus and, in case of etching, the distance between the etching lines.

3.6 Vector Network Analyzer

Vector Network Analyzers (VNA) are used to characterize high-frequency passive and active devices in their linear mode of operation by measuring their network parameters (S-parameters) as a function of the frequency. The S-parameters express the transmission (S_{ij} with $i \neq j$) and the reflection (S_{ii}) of a signal between two ports. A VNA measures both the phase and magnitude of the signal as well as the voltage and current in order to express the transmission and reflection coefficients [38]. In this work, it has typically been used to make transmission and reflection measurements in the range of microwaves. The VNA used at UCL is placed near to an electromagnet in order to allow characterization of a device under the influence of a magnetic field (see figure 3.7).

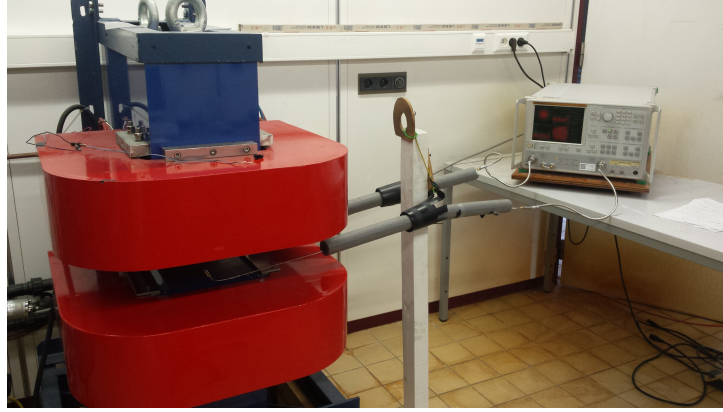


Figure 3.7: Picture of the VNA used in UCL. It is placed close to the red electromagnet in order to allow measurements under a magnetic field

In practice, the electromagnetic is linked to a power supply in order to apply a magnetic field on the device under test (DUT). A current supply allows the Hall sensor to measure the magnetic field that is reported via a multimeter to a computer. The RF signal is injected in the DUT using the VNA that can also treat the output signal. The VNA is also controlled by the computer. A scheme of the setup is shown in figure 3.8.

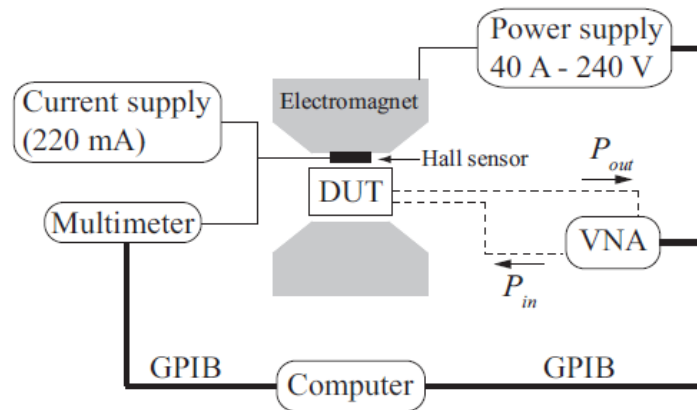


Figure 3.8: Schematics of the experimental setup for the FMR measurements, where the sample is represented by the Device Under Test (DUT). Thick lines represent GPIB cables, thin continuous lines represent electrical connections and the dashed lines represent cables for the rf-signal [7].

Two measurement modes exist: a sweep of frequency is done for each specified applied magnetic

field or a sweep of magnetic fields is done for each specified frequency. For the measurements of ferromagnetic resonance that interest us, the sweep of frequency allows to identify the ferromagnetic resonance frequency (maximum of absorption) for each applied magnetic field while the sweep in magnetic field gives the field at which a given frequency is the resonance frequency.

3.7 Alternating gradient magnetometer

The alternating gradient magnetometer (AGM) is used to measure the magnetic properties of a material. To allow this measurement, the magnetic material is attached on a cantilevered rod incorporating a piezoelectric element. Then, the sample is magnetized thanks to a DC magnetic field (variable in magnitude) and a simultaneous small alternating magnetic field gradient is applied. A force f appears following the formula

$$f = \nabla(M \cdot B) \quad (3.2)$$

where M is the amplitude of the magnetization of the sample and B the applied magnetic field. This force is transformed in voltage by the piezoelectric probe and the measurement of this voltage leads to a knowledge of the magnetization of the sample at each DC magnetic field. This allows for example to obtain the hysteresis curves of ferromagnetic materials [4, 39]. A picture of the AGM used in UCL is shown in figure 3.9.

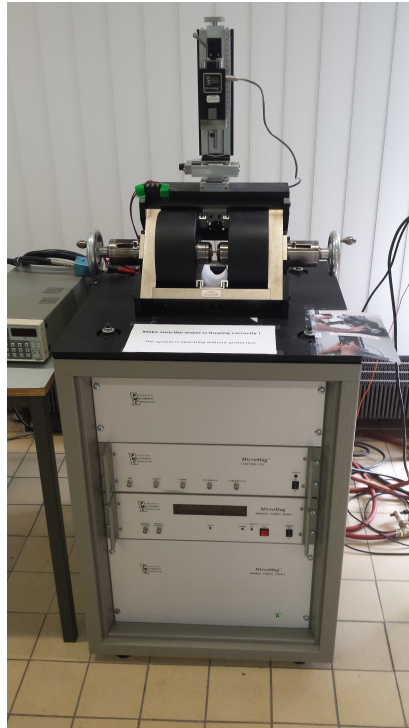


Figure 3.9: Picture of the AGM used in UCL.

3.8 Pseudo four-terminal sensing

The 4 terminal sensing is a technique used to measure the electrical resistance of low-resistance materials. Indeed, in order to measure the resistance of a material, it is necessary to measure a current flowing through it and the associated voltage in order to deduce the resistance using Ohm's law ($V = RI$). But if both intensity and voltage are measured using the same 2 contact

wires, the resistance of the wires is taken into account and is not negligible when the materials measured has low resistance. By using four contacts, it is possible to connect separately the wires of the current source and the wires for the potential measurements. The resistance of the probes used for voltage sensing is negligible since negligible current flows in these probes. This allows to have a good voltage measurement and hence a good resistance measurement.

When working with nanowired membranes, it is impossible to connect the four probes directly on one nanowire due to the small size and inaccessibility. Hence, we use a pseudo four-terminal sensing. In this case, the voltage measurement is done by placing one wire in contact with the ground plane of the membrane and the other with the part of the nanowires appearing when the nanowires overfill the membranes (their "head" is visible on top of the membranes; generally multiple nanowires are connected through their heads). The current is applied via one wire also connected to the work plane and the other wire also connected to the heads. This leads to a measurement of the resistance of the few nanowires connected together through their heads. A scheme of the setup is shown in figure 3.10.

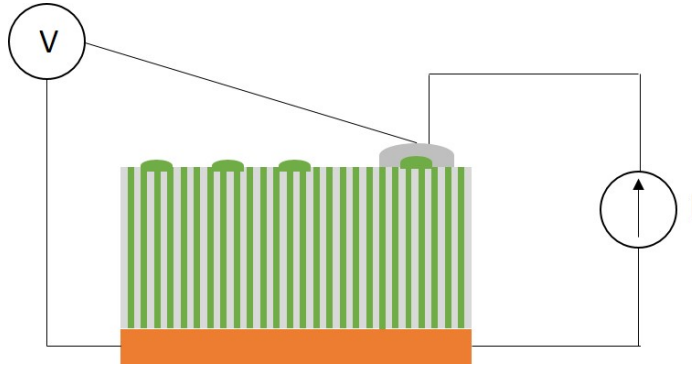


Figure 3.10: Scheme of a pseudo 4 terminal sensing with the cathode in orange, the alumina membrane in light grey, the nanowires in green, the metallic paste used for contacts in dark grey, a current source I and a voltmeter V .

Chapter 4

Synthesis, characterization and filtering properties of arrays of NiCo nanowires

This part in the work realized for this master thesis focuses on the increase of FMR absorption but with different materials. The first goal of this study is to characterize and compare the magnetic properties of nanowires produced by 2 solutions of NiCo leading to nanowires with a greater nickel ratio or with a greater cobalt ratio. This is done via AGM and RF measurements. The second goal is to focus on the filtering capabilities of these nanowires through FMR measurements and the general influence of the length of the nanowired zone and the average height of the nanowires on the absorption of a microwave signal.

Note that Sy and SM were used in these experiments. The Sy were first used because the available SM needed to be cut in smaller pieces by a picosecond laser in order to be usable. Once the SM were cut thanks to lasers from the industry Optec, they were used rather than Sy in order to allow the deposition of longer zones of nanowires.

4.1 The electrolytic solutions

The concentrations of the different components of the two electrolytic solutions are given in Table 4.1.

	NiSO ₄ , 6H ₂ O [M]	CoSO ₄ , 7H ₂ O [M]	H ₃ BO ₃ [M]	pH	E [V]
High nickel content	1	0.05	0.5	2.1	-1
High cobalt content	1	0.2	0.5	2.1	-1

Table 4.1: Concentration of the different compounds in the solution, pH of the solution and potential E° used for the electrodeposition of this solution.

The two solutions are exactly identical except for the concentration of the cobalt-containing compound. The EDX analysis of the nanowires deposited with these solutions and these parameters showed the proportions of nickel and cobalt in the composition of both types of nanowires:

- Nanowires deposited from **high nickel** concentration solution: 85% of Ni, 15% of Co
- Nanowires deposited from **high cobalt** concentration solution: 55% of Ni, 45% of Co

The first kind of nanowires will be referred to as "high nickel content" and the second one as "high cobalt content" even if it does not represent half of the composition. It is also interesting to note that the nanowires seemed to have a greater nickel proportion at the extremity on the cathode than at the other extremity (up to 10% more).

4.2 Magnetic characterization and filtering capabilities of the NiCo nanowires

4.2.1 Filling of the membranes

The characterization of the NiCo nanowires (for both high nickel and high cobalt contents) was performed on Sy and SM. Tests were first performed in order to determine the total charge to deposit in order to overfill the membranes (for Sy) or the total time needed for overfilling (for SM). The difference in the choice of the selected quantity to determine the moment of the overfilling of the membrane was linked to the irreproducibility of the depositions that will be discussed in the section devoted to this subject.

Using the data extracted from these tests, depositions were first done on Sy. The targeted height of the nanowires was around 80 – 90% of the height of the membrane ($100\mu m$). However, the obtained height was in fact much lower. The nanowired Sy selected for the general magnetic characterization had zones of $5.9mm$ of diameter deposited with NiCo nanowires showing an average height of the nanowires close to $60\mu m$ for the high nickel content nanowires and close to $50\mu m$ for the high cobalt content nanowires, as can be seen on the pictures obtained by optical microscopy shown in figures B.3 and B.4.

In order to have a good comparison with the NiCo nanowires deposited in Sy, SMs with similar nanowire filling were chosen. The filling of the high nickel content nanowires is also around $60\mu m$ while the filling of the high cobalt nanowires is closer to $55\mu m$ as it can be seen on figures B.7 and B.8.

4.2.2 AGM measurements

Comparison of high and low nickel content NiCo nanowires

AGM measurements were performed on the different membranes described here above. Let's first take a look at the hysteresis curves for the two nanowired Sy, represented on figure 4.1. Note that the CGS system is often used here rather than SI units because the instruments used to apply magnetic fields have output data using CGS units. It is then easier and logical to compare the results in this same system.

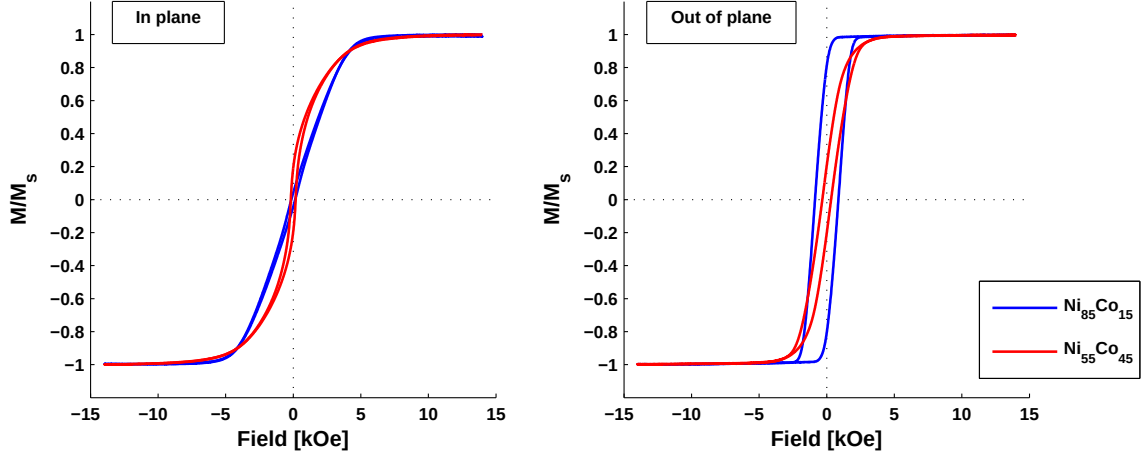


Figure 4.1: Hysteresis loops of the high nickel content and high cobalt content nanowires deposited on Sy with respective heights $61\mu m$ and $50\mu m$.

Results shown in [40] suggest similar behaviour of high nickel content NiCo nanowires and high cobalt content NiCo nanowires with respectively pure Ni and pure Co from a solution with pH=5. The AGM curves of pure nickel and pure cobalt pH=5 are shown in figure 4.2.

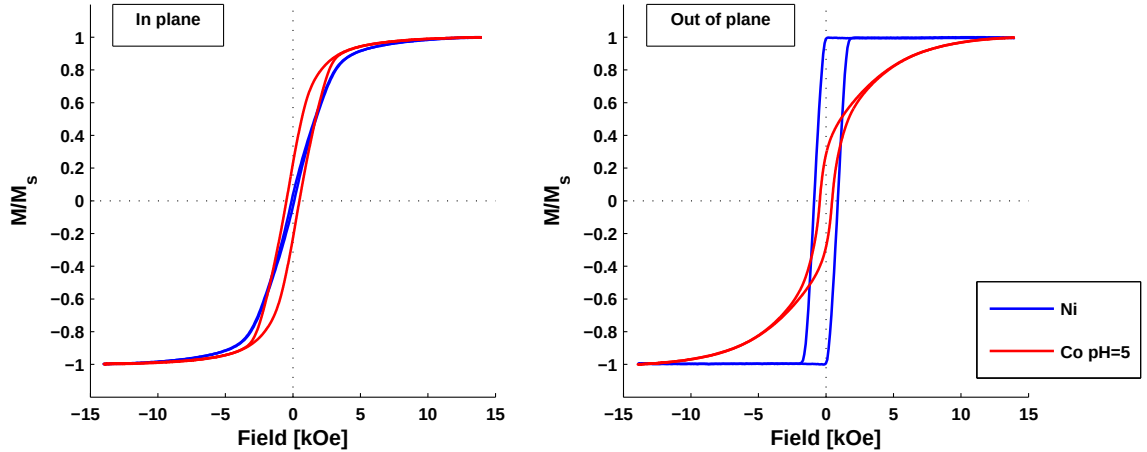


Figure 4.2: Hysteresis loops of pure Ni nanowires and pure Co nanowires deposited from a solution of pH=5 on SM.

It can be seen that the NiCo with high nickel content is very similar to the pure nickel. The one with high cobalt content shows more intermediate curves between the pure Ni and the pure Co deposited from a solution with pH around 5. In this condition, the Co shows a predominant hcp structure with the c-axis oriented perpendicular to the axis of the nanowires with still some fcc crystals. This induces a magnetocrystalline anisotropy contribution perpendicular to the magnetostatic one. Hence, the total anisotropy is the result of the subtraction of the magnetocrystalline anisotropy contribution to the magnetostatic contribution. It seems to also be the case for high cobalt content NiCo too but with a lower magnetocrystalline anisotropy and therefore probably a lower fraction of hcp crystals.

We can see on the curves for NiCo that the anisotropy is still predominantly parallel to the axis of the nanowires for both types. Indeed, the hysteresis curves with a magnetic field in

plane, i.e. in the plane of the membrane and hence perpendicular to the axis of the nanowires, show low remanence. On the contrary, with an out of plane magnetic field, the remanence is important. This means that when a nanowire is magnetized along its axis, it will tend to stay in this position, it is the easy axis and hence it is difficult to overcome the anisotropy field in this direction to reach the other easy direction (also along the axis of the nanowire but with the magnetization in the other direction). It is however much easier for the magnetization of the high cobalt content to reverse because of the magnetocrystalline anisotropy contribution perpendicular to the axis. The magnetostatic anisotropy contribution still seems to outmatch the magnetocrystalline anisotropy contribution since the easy axis is parallel to the nanowires axis. With a magnetic field perpendicular to the nanowires, no global anisotropies stabilize the magnetization and hence no or almost no remanence can be observed.

Finally, from the straight shape of the curve, we can state that the high nickel content NiCo nanowires behave almost as ideal single magnetic domain particles or so-called Stoner–Wohlfarth particles (similarly to the pure Ni). The rotation of the magnetization is then coherent, i.e. magnetic moments in a single magnetic domain rotate in unison. This is however not the case for high cobalt content NiCo and pure Co nanowires [4,41]. They need to be saturated in order to behave like single-domain particles.

Comparison Sy and SM

We can compare the hysteresis curves in Sy with the curves obtained for the depositions in SM with similar filling. The hysteresis curves for high nickel content in Sy and SM are represented on figure 4.3 and the one for low nickel content in Sy and SM can be seen on figure 4.4.

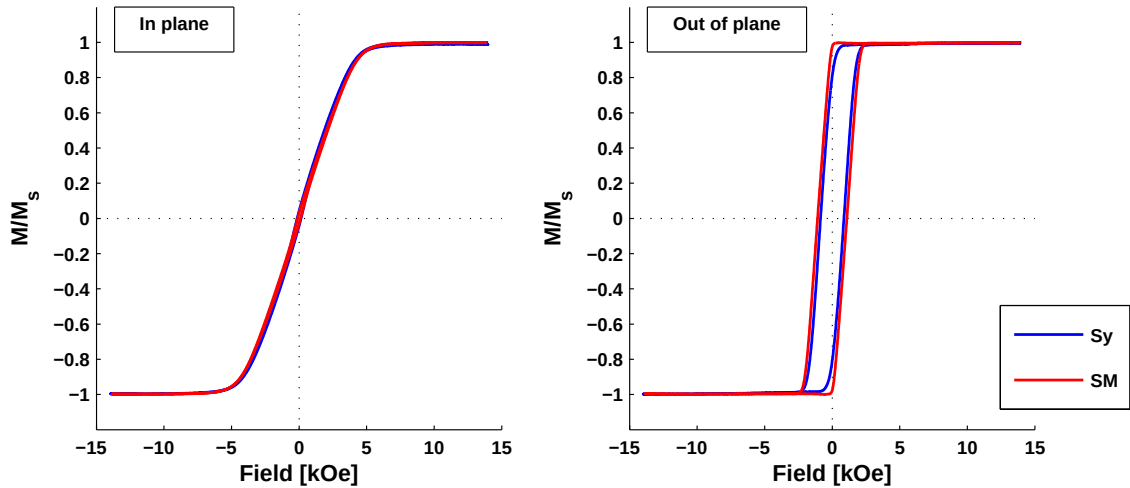


Figure 4.3: Hysteresis loops of the high nickel content nanowires deposited in Sy and SM with similar filling (around $60\mu m$).

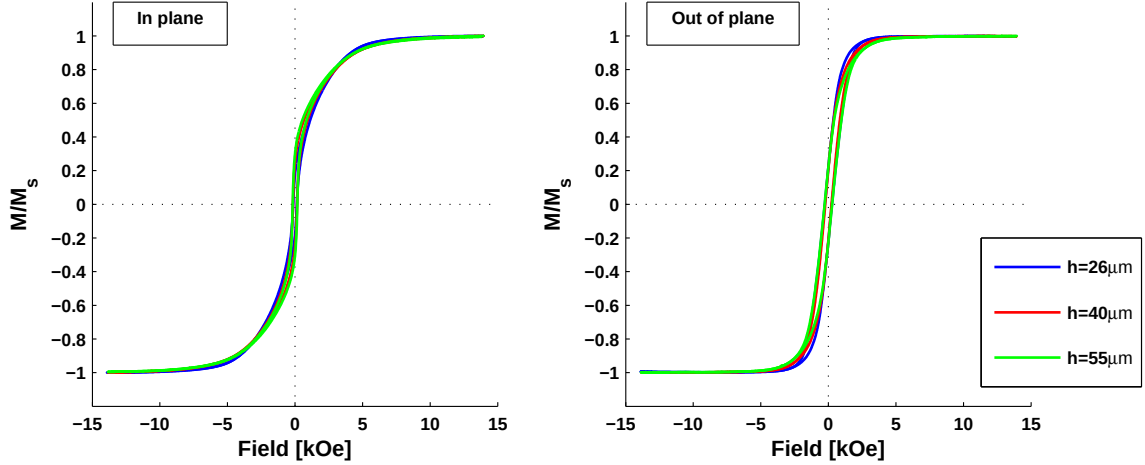


Figure 4.4: Hysteresis loops of the high cobalt content nanowires deposited in Sy and SM with similar filling (around $50\mu m$)

We can see that the curves are logically very similar for Sy and SM. The small difference can be explained by the difference in pore size ($35nm$ in Sy and $40nm$ in SM) and mostly the difference in porosity (12% in Sy and 10% in SM). Since Sy has a larger porosity, the magnetostatic anisotropies are smaller (see equation 2.20) and hence the coercive field is a bit smaller. The small differences in the shape can also be attributed to small variations in the cathode composition and structure, small changes in the positioning of the membranes on the AGM probes or a slight difference in the structure of the nanowires.

Approximation of the anisotropies

It is possible to provide a first approximation of the anisotropies for both high nickel content and high cobalt content nanowires using the anisotropy field distribution (AFD) method [42]. This method assumes an assembly of Stoner–Wohlfarth particles with parallel easy axis. It then defines $\sigma(H)$ the normalized probability of finding the value H for the anisotropy field. This probability can be computed using

$$\sigma(H) = -H \frac{d^2 m(H)}{d^2 H} \quad (4.1)$$

where $m(H)$ is in practice the descent of magnetization from saturation to the remanent state along the hard axis. The uniaxial anisotropy field H_a of a particle is then given by the field at which $\sigma(H)$ is maximum.

Since we have seen that high nickel content NiCo nanowires behaved more like Stoner–Wohlfarth particle but not the high cobalt content ones, this model is a priori adapted only for the first kind. It was also shown that the obtained value could be overestimated for nanowire diameters only slightly larger than the ones of the nanowires deposited in this work [41]. Hence, we can expect to have results just slightly overestimated.

The value of H_a has been computed using the in plane hysteresis curves for high nickel and high cobalt content on Sy. The uniaxial anisotropy field given by the AFD method is $3.87kOe$ for high nickel content nanowires and $3.31kOe$ for high cobalt content nanowires. Note that these values have been computed numerically with some smoothing methods in order to obtain usable data

and hence that some inaccuracies may have been introduced.

4.2.3 FMR measurements

Filtering capabilities

RF (radio frequency) measurements were done using the VNA and a magnetic field source in order to study the absorption of electromagnetic waves in the nanowired membranes. In the experiments done in this work, the magnetic field is always applied in the direction out of the plane of the membrane and hence parallel to the nanowires and the sample is always first magnetically saturated. Microstrip lines were deposited on the nanowired membranes in order to allow the propagation of the electromagnetic waves. The width of the microstrip lines was not adjusted to match the 50Ω transmission lines of the VNA for practical reasons. Indeed, it is difficult to do a mask for each membrane height used in the labs and it is difficult to connect very narrow microstrip lines. Furthermore, when doing a sweep in frequency, the "noise" due to the mismatch and the losses can be eliminated or at least reduced as it will be explained later on. Note that using a picosecond laser, it is possible to etch a metal layer deposited on top of the membranes in order to remove everything except a thin microstrip line with the width necessary for impedance matching. However, the laser had some issues at the time of these experiments. Furthermore, even if a precise microstrip line could be obtained, the problem of difficult connection with such thin lines would persist anyway. For sweeps in magnetic field, the curve is automatically less noisy.

Let's have a quick look at the transmission curve for the high nickel content nanowires on Sy as a function of the frequency with a strong applied field ($9000Oe$) and without (represented on figure 4.5).

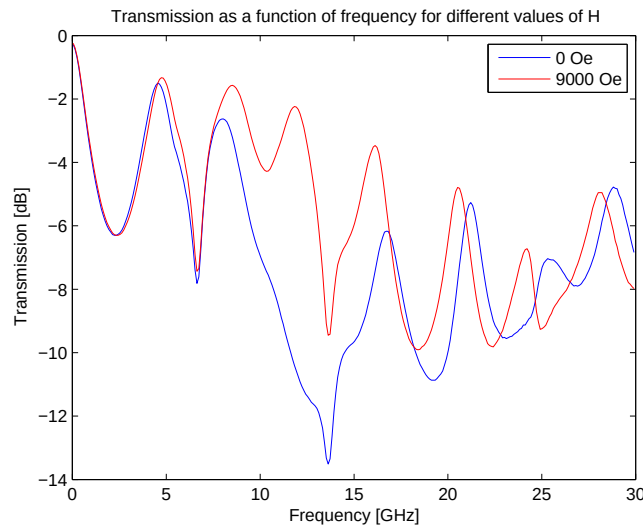


Figure 4.5: Transmission vs frequency curve of Sy charged with high nickel content nanowires of height $61\mu m$ on a zone of diameter $5.9mm$, with $H = 9000Oe$ and $H = 0Oe$.

The transmission is expressed in dB where $X_{dB} = 10\log(P_2/P_1)dB = 20\log(V_2/V_1)dB$ with X the transmission in dB , V_1 and P_1 respectively the output voltage and power and V_2 and P_2 respectively the input voltage and power. It is however more relevant to consider the expression with voltages when it is about FMR absorption since the absorption has a direct influence on the magnetic field of the electromagnetic wave and hence the electric field and the voltage.

We are interested in the absorption at zero applied magnetic field by the ferromagnetic nanowires. However, a lot of perturbations are induced by the mismatch of impedance and the losses in the microstrip line. At high field ($9000Oe$), the absorption peak appears at much larger frequencies than the peak at zero field (usually more than $20GHz$ further than the absorption peak at $0Oe$). Hence, only the perturbations due to the mismatch and the losses remain. They can then be subtracted to the curve at zero field in order to eliminate the perturbations. Note however that, since the transmission is expressed in dB, subtracting the curve at high field to the curve at low field is equivalent to express the transmission at low field relatively to the transmission at high field ($20\log(V_3/V_1) - 20\log(V_2/V_1) = 20\log(V_3/V_2)$ with V_1 the initial voltage, V_2 the output voltage at high field and V_3 the output voltage at zero field for one frequency). We can then observe the relative absorption of the nanowires in dB that, when converted in a linear scale, is equivalent to the percentage of absorption of the signal being transmitted despite the losses (signal at high field). Since the losses are not constant on the whole range of frequencies, it means that we can't compare two curves at different frequencies in term of absolute absorption. Indeed, even if, after subtraction, the two absorption curves have the same minimum value, if the losses at the two frequencies were not equal, the absorbed power is not the same since we then have the same percentage of absorption but of two different voltages (output voltages at high field for these two frequencies). In order to obtain an absolute value of absorption, the subtraction should be made in linear scale (keeping the same V_1 , V_2 and V_3 as before, we then have $V_2/V_1 - V_3/V_1 = (V_2 - V_3)/V_1$). Then, we would obtain the percentages of absorption relatively to the initial input voltage, which is the same at all frequencies.

It is not clear whether the most logical representation is the absolute or relative absorption. An experiment that could be done is doubling the input voltage and then observe the transmission. If the relative absorption is the same, it would mean that the nanowires absorb a fixed proportion of the signal they are able to intercept and hence the relative absorption would be the most logical since the input value is not relevant. If the absorption is twice as small, it means that the nanowires are able to absorb a fixed power and hence the absolute absorption is the most logical to represent since we have to keep the input value in the result. Nevertheless, we should expect a mix of the two. Since the nanowires are not doing the whole height of the substrate, they will only be able to intercept a fixed proportion of the signal. But we cannot expect them to absorb infinite amount of energy. Varying the input power could then help find a global definition of the way the nanowires absorb the signal. But for the moment, we are going to subtract in dB and hence describe the relative absorption of the ferromagnetic nanowire.

We can see that the transmission is very noisy and that the two curves never join even though the perturbations should be the same at low and high field away from the peaks. It is probably due to unstable sample connections. Hence, the noise can't be exactly canceled, but the curve resulting from the subtraction of the high field curve to the low field curve still clearly shows the absorption peak as seen on figure 4.6 (a). The positive values of transmission have no physical meaning and are only induced by the imprecise noise subtraction. We can observe an absorption around $8dB$ which already proves the filtering capabilities of these magnetic nanowires even without any external magnetic field.

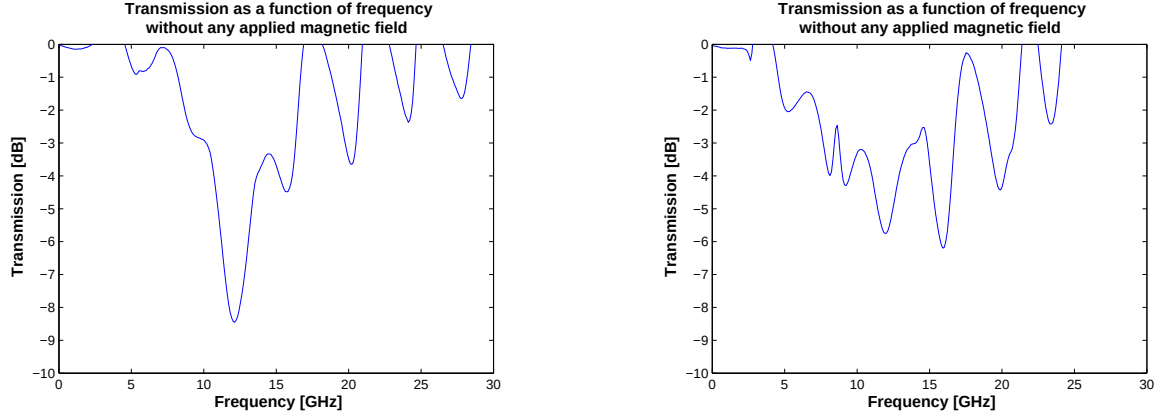


Figure 4.6: Transmission vs frequency curve for Sy membranes without any applied magnetic field after noise subtraction, charged with (a) high nickel content nanowires of height $61\mu\text{m}$ on a zone of diameter 5.9mm and (b) high cobalt content nanowires of height $50\mu\text{m}$ on a zone of diameter 5.9mm .

The curve computed using the same principle for high cobalt content nanowires is represented on figure 4.6 (b). Here, we can observe a small absorption peak even broader and shallower at the same time, which makes difficult the precise identification of the frequency corresponding to the maximum of absorption i.e. the ferromagnetic resonance frequency. The maximum of absorption is around a few dB ($5 - 6\text{dB}$). It shows that this material is less interesting for filtering applications, which seems logical since the nanowires are not magnetically saturated at zero field. The broadness of the peak could be attributed to the overlap of two absorption peaks. Indeed, since the high cobalt content NiCo nanowires are closely related to pure Co nanowires at pH around 5, it means that there are probably fcc and hcp crystals in the structure. Since fcc crystals show negligible magnetocrystalline anisotropy but hcp crystals show a non-negligible contribution, this would lead to 2 distinct absorption peaks [9]. The noise and a large Gilbert damping coefficient could explain why the 2 peaks are not clearly distinguishable since the broadness of the peak can be linked to the damping coefficient α by the relation

$$\Delta H = \Delta H_0 + 1.16\alpha\omega/\gamma \quad (4.2)$$

with ΔH_0 a frequency-independent contribution to the linewidth, ω the angular frequency and γ the gyromagnetic ratio.

Magnetic characterization

The same measurements were done for the SM but measurements were also done with a magnetic field sweep in order to obtain more accurate values of the resonance frequency. The resulting curves with transmission expressed in arbitrary units are shown in figure 4.7.

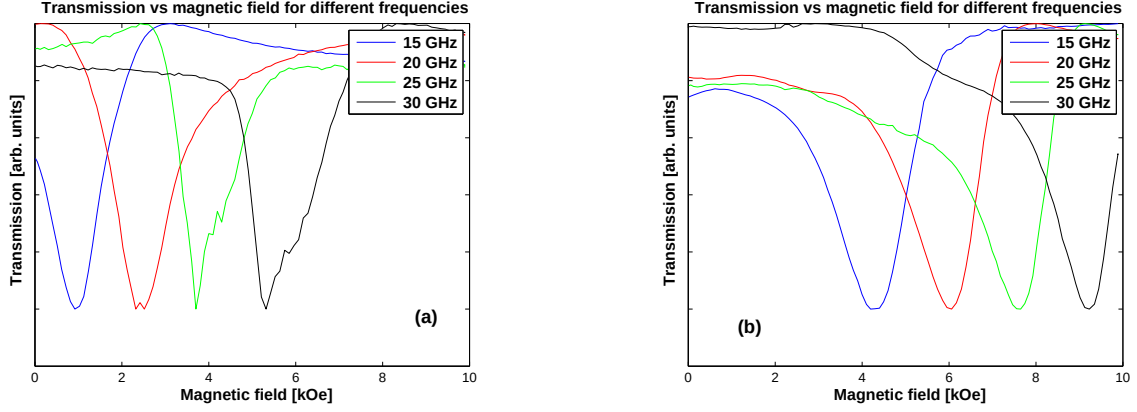


Figure 4.7: Transmission (in arbitrary units) as a function of the magnetic field for different microwave frequencies (15GHz, 20GHz, 25GHz, 30GHz) for high nickel content nanowires (a) and high cobalt content nanowires (b) in SM with respective average heights $59\mu m$ and $55\mu m$ on zones of respective lengths $1.1cm$ and $0.7cm$.

From these data, it is possible to extract the value of the gyromagnetic ratio and the anisotropies. Indeed, equation 2.53 showed that $\omega_{FMR} = \mu_0\gamma H$ in the International System of Units. Hence,

$$f_{FMR}^{SI} = \frac{1}{2\pi} \mu_0 \gamma^{SI} H^{SI} \quad (4.3)$$

Since we have the results in CGS system, it is easier to use the relation in this system:

$$f_{FMR}^{CGS} = \frac{\gamma^{CGS}}{2\pi} H^{CGS} = \frac{\gamma^{CGS}}{2\pi} (H_{app}^{CGS} + H_{ani}^{CGS}) \quad (4.4)$$

Redrawing the previous curves as resonance frequency vs applied field curves will allow to deduce a value of γ to a factor 2π . The values obtained at constant field and at constant frequency are all used even if the latest are supposed to be more precise because of the smaller noise. The resulting curves are shown on figure 4.8:

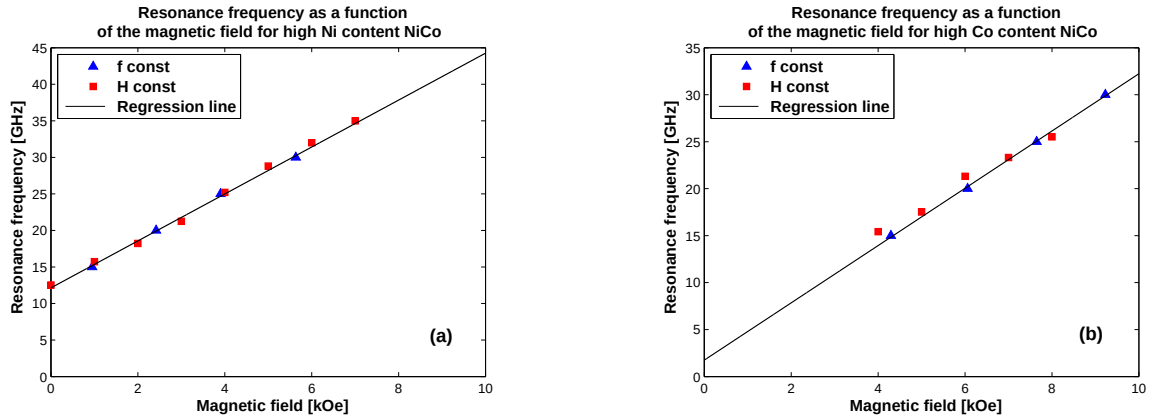


Figure 4.8: Resonance frequency as a function of the applied magnetic field for constant frequency mode (triangles) and constant magnetic field mode (squares) for high nickel content NiCo nanowires (a) and high cobalt content NiCo nanowires (b) in SM with respective average heights $59\mu m$ and $55\mu m$ on zones of respective lengths $1.1cm$ and $0.7cm$.

Note that on the curve for high cobalt content, the values at fields below $3kOe$ are not represented because the sample is not at saturation anymore and since its nanowires do not behave as single

magnetic domains, when the saturation is lost, the model described cannot be applied to it. Hence, the results will not be coherent with the results at higher fields and will not help to gather any information.

The saturation magnetization of the NiCo can be estimated to vary linearly between the magnetization saturation of pure Ni ($485 * 10^3 A/m$ [7]) and hcp cobalt ($1370 * 10^3 A/m$ [7]) with the proportion of nickel. Hence, the approximation of the saturation magnetization of high nickel content NiCo is $M_s = 618 * 10^3 A/m$ and the one for high cobalt content NiCo is $883 * 10^3 A/m$.

The slope and the frequency at zero field of the regression lines obtained by the method of least squares allow us to extract the values of the gyromagnetic ratio and of the anisotropy field.

For high nickel content membranes, the slope is $3.21 GHz/kOe$. Since the slope is equal to $\gamma/2\pi$, $\gamma_{CGS} = 20.18 GHz/kOe$. Converting back in SI units, we have $\gamma_{SI} = 201.8 GHz/T$ corresponding to a g-factor worth ~ 2.3 . The anisotropy field found by dividing the resonance frequency at zero field by the slope is $3.78 kOe$. This value is close to the one obtained via the AFD method and just a bit smaller, as expected. Using the approximation of the saturation magnetization given before, the magnetostatic anisotropy contribution can be estimated using $H_{ms} = 2\pi M_s(1 - 3p) = 2.72 kOe$ which is smaller than the obtained anisotropy field. This difference can be explained by a magnetoelastic contribution of the order of $1 kOe$. Indeed, this anisotropy has already been reported for pure Ni nanowires in anodized alumina membranes [43]. Thus it wouldn't be surprising to find the same type of contribution in high nickel content materials.

For high cobalt content membranes, the slope found is $3.05 GHz/kOe$. Hence, $\gamma_{CGS} = 19.15 GHz/kOe$ and $\gamma_{SI} = 191.5 GHz/T$ for a corresponding Landé factor of $g = 2.18$. The anisotropy field is worth $0.57 kOe$ which is very different from the one estimated by AFD. It proves that the high content NiCo nanowires are indeed not ideal Stoner-Wohlfarth particles. The value of the anisotropy is quite weak, showing that the magnetocrystalline anisotropy contribution perpendicular to the axis of the nanowires is not negligible. The magnetostatic anisotropy contribution can be estimated as before using the approximated saturation magnetization: $H_{ms} = 2\pi M_s(1 - 3p) = 3.88 kOe$. Since no substantial magnetoelastic anisotropy has been reported in *Co* nanowires, we can expect it to be quite weak. The magnetocrystalline anisotropy contribution can then be estimated to lie around $3.3 kOe$.

The values found for the Landé factors of both types are quite high but still similar to the ones of pure Ni ($g = 2.2$) and hcp Co ($g = 2.18$) [7].

A summary of the results and the comparison with pure Ni and pure Co deposited from a solution with pH=5 is shown in table 4.2.

	High Ni content NiCo	high Co content NiCo	pure Ni	pure Co pH=5
$\gamma[GHz/kOe]$	20.18	19.15	19.41 [6]	19.16 [13]
g-factor	2.3	2.18	2.2	2.18
$M_s[emu \cdot cm^{-3}]$	618	883	485	1370
$H_{ani}[kOe]$	3.78	0.57	2.13	0.5 – 1 [9]

Table 4.2: Comparison of the gyromagnetic ratio (γ), g-factor, saturation magnetization (M_s) and total anisotropy field (H_{ani}) for the high nickel content and high cobalt content NiCo nanowires studied and pure Ni and pure Co nanowires (from a solution with pH=5) deposited in nanoporous alumina membranes.

4.3 Double absorption peaks

In 2010, Medina et al. showed double absorption peaks when an array of nanowires went from saturation state to a state close to the zero magnetisation (around the coercive field) [44]. It was shown that at magnetic field values around the coercive field, nanowires are still saturated but in the 2 opposite directions along their axis which leads to two distinct absorption peaks (let's note the magnetic moments of the nanowires m_+ when oriented in the same direction as positive H and m_- when oriented in the other direction). In fact, since the applied magnetic field is oriented parallel to the magnetization of some nanowires and antiparallel to the magnetization of others, the resonance frequencies $f_{m_{\pm}}$ corresponds to

$$f_{m_{\pm}} = \gamma\mu_0(\pm H_{app} + H_{ani}) \quad (4.5)$$

Note also that the value of H_{ani} is more complicated than described before because of the opposite magnetizations in the array of nanowires. The two peaks join each other when going back to zero applied magnetic field. Without any applied magnetic field, the nanowires are still saturated in two different directions but since the applied field is zero, the resonance frequency is the same for both. To be precise, the applied field at which the peaks join each other is not exactly zero if we haven't decreased the field to the exact value of the coercive field because then $m_+ \neq m_-$ which leads to differences in the dipolar interactions for both types [7].

In order to observe the double peaks of absorption, an estimation of the coercive field of the membranes for both high nickel and high cobalt content nanowires has to be performed. They can be extracted from the out of plane hysteresis loops shown in figure 4.1. The coercive field (when coming from positive saturation) is around $-860Oe$ for the membrane with high nickel content nanowires and around $-330Oe$ for the membrane with high cobalt content nanowires.

The double peaks of absorption have been observed on Sy with a zone of $1cm$ of nanowires deposited. The nanowired zone on this membrane being longer, the depth of the peaks is greater and it is therefore easier to identify these peaks than on the previous samples. While the double peaks are clearly visible with the high nickel content nanowired zones, they could not be identified with high cobalt nanowires. This can be associated once again to the fact that high cobalt content NiCo nanowires are not single magnetic domains under $3kOe$. Hence, the nanowires are not anymore saturated in two different directions around the coercive field which prevents the apparition of the double absorption.

The double peaks for high nickel content nanowires at nonsaturated field values $H = -1000Oe$, $H = -500Oe$ and $H = 0Oe$ are shown in figure 4.9.

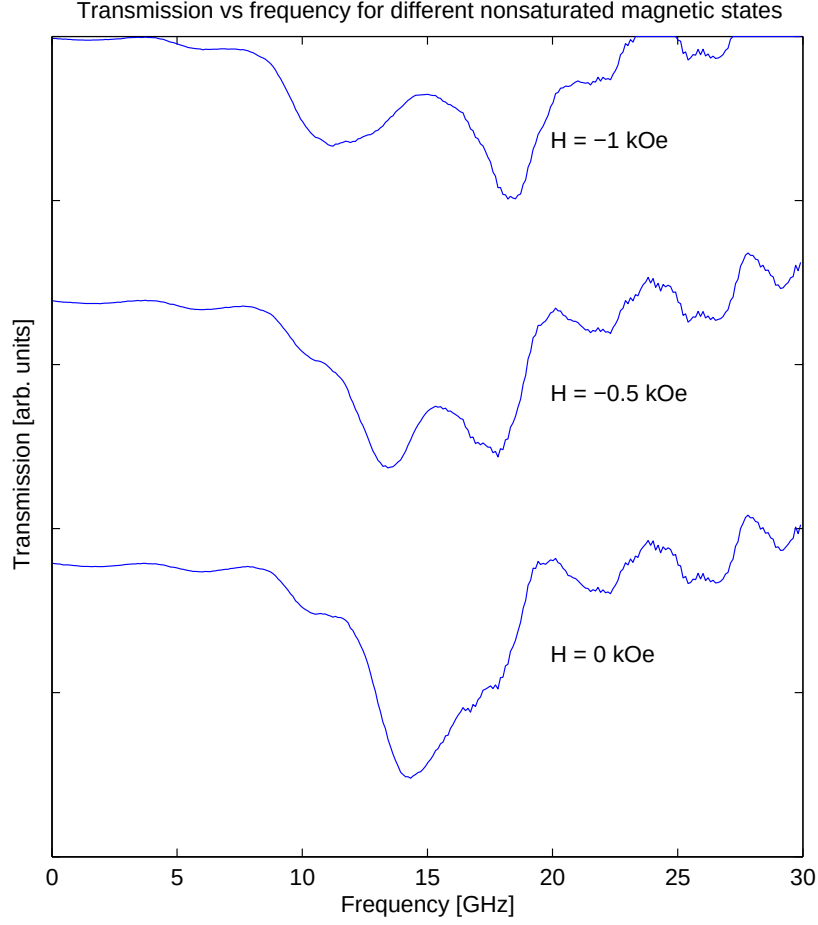


Figure 4.9: Transmission in arbitrary units vs frequency for a Sy containing high nickel content nanowires with a height of $30\mu m$ deposited on a zone of $10mm$ of diameter, for nonsaturating magnetic fields $H = -1000Oe$, $H = -500Oe$ and $H = 0Oe$.

Since the magnetic field has been decreased from saturation to a value under the coercive field, there should be a greater fraction of m_- than m_+ . It is indeed what we observe since the peak at higher frequency for $H = -1kOe$ is deeper. Since the applied field is negative, the largest value of the resonance frequency is obtained for the nanowires magnetized in the negative directions. Therefore, we can say that the deepest peak is indeed associated to m_- . When the magnetic field goes up, the peaks shift toward each other and join. We see that at $H = 0kOe$, the peaks are not exactly joined, as expected.

This once again proves the ideal single domain nature of the high nickel content NiCo nanowires.

4.4 Magnetic field influence on FMR absorption

It can be seen from the formula of the ferromagnetic resonance frequency $f_{FMR} = \frac{1}{2\pi}\gamma\mu_0(H_{app} + H_{ani})$ that the applied magnetic field has a direct influence on the ferromagnetic resonance frequency. This relation has been used in order to identify the anisotropies and the gyromagnetic ratio. It is interesting to check if it is the only influence on the FMR absorption. The differences of the absorption peak can be clearly seen on figure 4.10 representing the transmission of a microwave signal in a high nickel content nanowired zone of $10mm$.

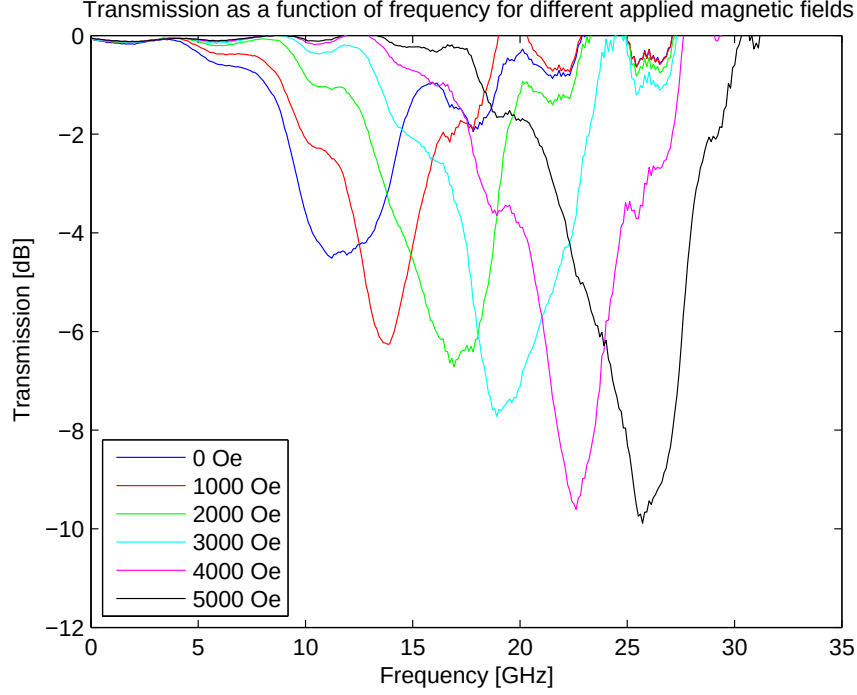


Figure 4.10: Transmission vs frequency for a Sy containing high nickel content nanowires of height $61\mu m$ on a zone of diameter $5.9mm$, for different applied magnetic fields $H = 0Oe$, $H = 1000Oe$, $H = 2000Oe$, $H = 3000Oe$, $H = 4000Oe$ and $H = 5000Oe$.

With the increase in frequency, there is an increases in the absorption peak in dB but also the noise. The absorption increases faster than the noise in dB, leading to the differences in height at different field values. We can switch back from the transmission S_{21} in dB to the transmission T_{21} in linear scale using the reciprocal function of the one used by the VNA to convert in dB:

$$S_{21} = 20\log(T_{21}) \Leftrightarrow T_{21} = 10^{S_{21}/20} \quad (4.6)$$

The resulting transmission is shown in figure 4.11.

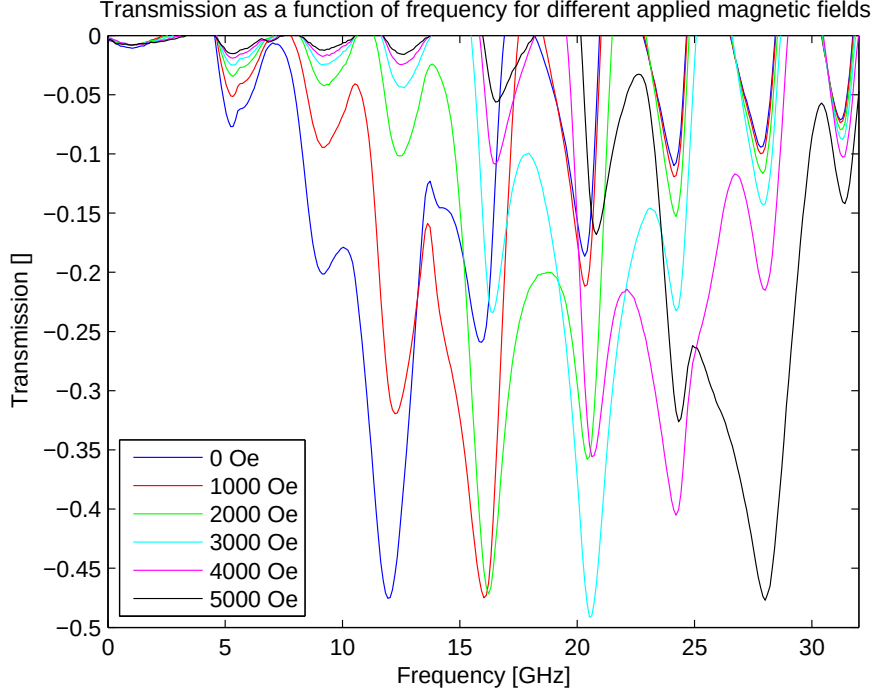


Figure 4.11: Transmission vs frequency in linear scale for a Sy containing high nickel content nanowires of height $61\mu m$ on a zone of diameter $5.9mm$, for different applied magnetic fields $H = 0Oe$, $H = 1000Oe$, $H = 2000Oe$, $H = 3000Oe$, $H = 4000Oe$ and $H = 5000Oe$.

Here, we can see that besides the switch in frequency, the magnetic field has no effect on the power absorbed. The unclear peaks or strange shifts in frequency are associated to unprecise noise substraction. Hence, we have similar absolute absorptions but different relative absorptions. Since the sample keeps a saturation magnetization almost until zero applied magnetic field, we would expect the absorption to stay the same. This is then an argument in favor of the use of the absolute absorption. In order to check that the absorption must indeed be similar at all fields, the same experiment should be done with a microstrip line matching the impedance of the transmission line or even in a waveguide matched to the transmission line. The losses would then be weak enough to observe in both dB and linear scale if the peaks have the same height or not.

4.5 Length influence on FMR absorption

One of the goals of the multiple depositions on Sy was to observe the influence of the length of the nanowired zone on the absorption and see if a relation could be deduced. As can be seen on figure 4.12 for the high nickel content nanowired membranes, the noise is too high to obtain values accurate enough for this purpose. Furthermore, due to highly irregular filling of the membranes, the height of the nanowires highly varies preventing isolation of the length influence. Note that this should be investigated for both relative and absolute absorptions. However, the high perturbations in the signal made it difficult to clearly observe the absorption in linear scale. Hence, the developments will only be performed here with the relative absorption, knowing that more experiments should be done in order to investigate the length influence on the absolute absorption too.

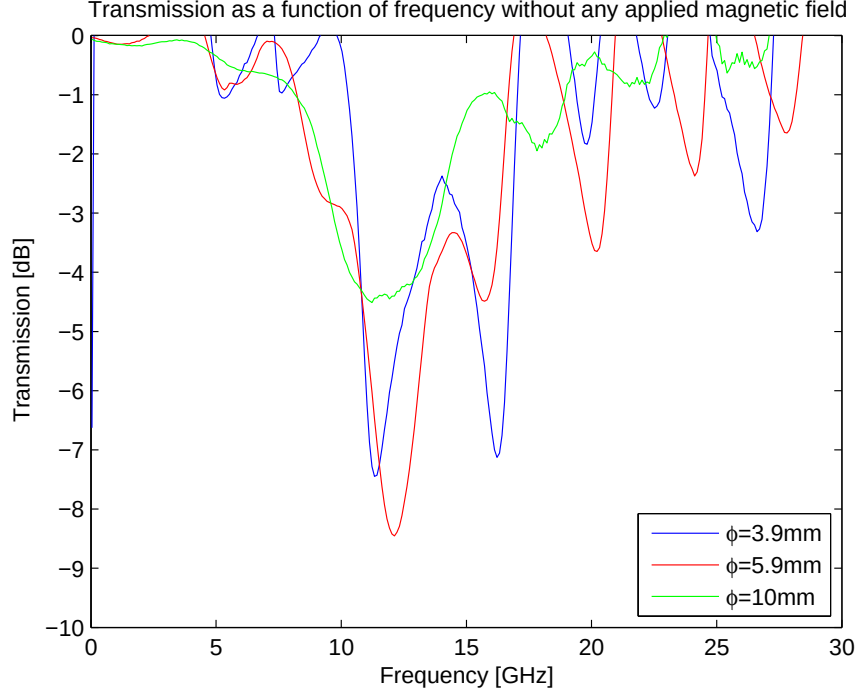


Figure 4.12: Transmission vs frequency for a Sy containing high nickel content nanowires for different diameters ϕ of nanowired zones traveled by the signal: $\phi = 3.9\text{mm}$, $\phi = 5.9\text{mm}$, $\phi = 10\text{mm}$ (respective heights are $77\mu\text{m}$, $61\mu\text{m}$ and $30\mu\text{m}$).

It is even worse for high cobalt content nanowires and hence not interesting to show. It is logical to expect an increase of the power absorption for longer propagation of the electromagnetic waves in the nanowired medium since each nanowire takes part to the absorption. Hence, more nanowires would logically mean more absorption. However, we can not expect a linear dependence between length and absorption in linear scale since the signal power at the resonance frequency will decrease and less power can thus be absorbed further from the electromagnetic waves source. But we could expect a linear dependence in dB (corresponding to a fast increase in absorption at small lengths and an asymptotic dependency tending to 100% absorption for longer nanowired zones).

We can analyze the figure 4.13 comparing Sy and SM filled with nanowires of same composition and similar heights. The difference in absorption cannot be attributed to the small changes between Sy and SM and are hence clearly due to the change in length. The increase in absorption in high nickel content is comparable to the one for high cobalt content with respect to the changes in length. The ratio of the maximum of absorption for different lengths of nanowired zones and the ratio of the lengths are respectively 6.5 and 5 for high nickel content nanowires and 2.9 and 2.4 for high cobalt content nanowires). These differences could be attributed to the important perturbations. This suggests that the absorption scales more or less linearly with the length of the nanowired zone but it should be checked with more data.

Besides these considerations, note that the absorption obtained with a long zone of high nickel content NiCo nanowired is over 40dB (originally 60dB with under 20dB of noise) which corresponds to an absorption greater than 99% of the power of the signal at this frequency. This proves the suitability of nanowire arrays for filtering applications. Note however that this result has only been reproduced at low fields but the absorption (in both dB and linear scale) decreases at high field. Since it has been shown that H should not decrease the power absorption, further

investigations should be performed in order to reproduce these results.

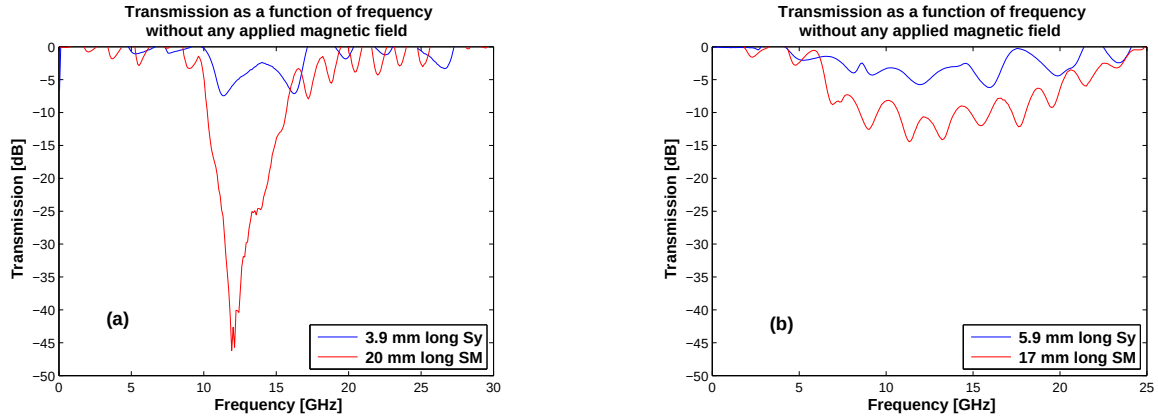


Figure 4.13: Transmission vs frequency at zero magnetic field for (a) 3.9mm long Sy and 20mm long SM with similar filling of high nickel content nanowires around 80% and (b) 5.9mm long Sy and 17mm long SM with similar filling of high cobalt content nanowires around 50%.

4.6 Height influence on magnetic properties

Let's now try to get some hints about the influence of the height of the nanowires on the magnetic properties of the membranes. Different SM were filled with different heights of nanowires in order to allow the comparison. The nanowired zones of the membranes were supposed to be about the same length but due to practical issues like breaking of membranes or size adjustment in order to allow connections, various membrane lengths have been used. We can however try to compare membranes with similar lengths and different pore fillings. A comparison for high cobalt content nanowired membranes with comparable nanowired lengths (L) and different fillings (h) can be seen on figure 4.14. Note that the height of the nanowires is not homogeneous in the whole membrane as can be seen for example for the SM membrane of length 25mm containing high nickel content nanowires (see pictures B.9, B.10 and B.11). The heights of the nanowires at three different spots of the membranes are indeed quite different (heights are $68\mu m$, $95\mu m$ and $79\mu m$). Hence, the values given for the height of the nanowires are the results of an averaging of the filling at the extremities and the middle of the membrane.

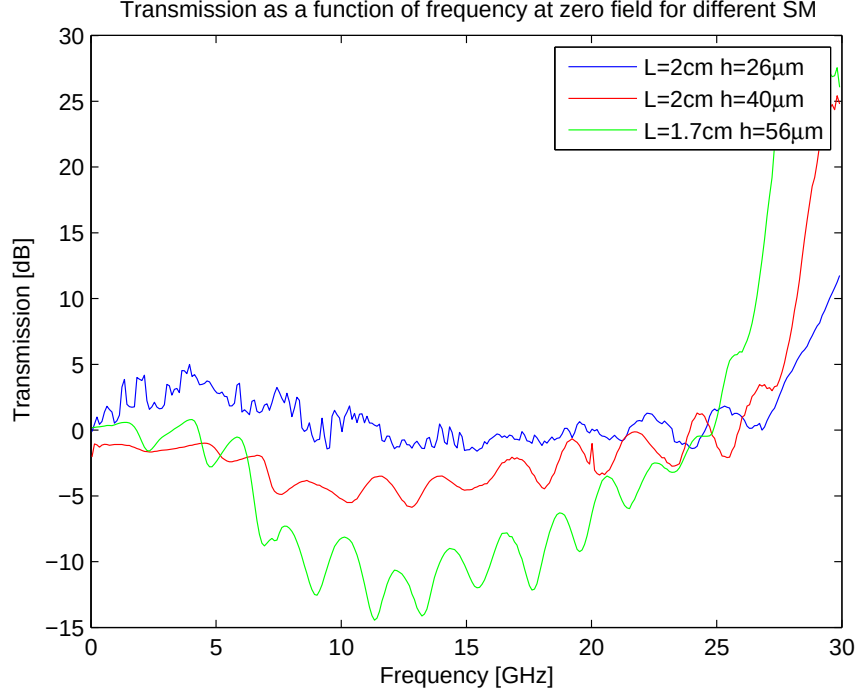


Figure 4.14: Transmission vs frequency at zero field for different SM with high cobalt content nanowires of length L and nanowires height h .

We can see that although the membrane is long, the one filled with $26\mu\text{m}$ of nanowires show almost no absorption. The second one with the same length but $40\mu\text{m}$ high nanowires show observable absorption and the third one with $56\mu\text{m}$ high nanowires show even more absorption despite a slightly shorter deposited zone. This clearly shows the influence of the height of the nanowires that can be associated to a better interception of the microwave signal. Indeed, nanowires filling only a small portion of the membrane will let an important part of the signal pass without being able to intercept it. Letting the nanowires grow more allow to intercept a larger part of the signal and hence absorb more power.

The hysteresis loops of the membranes with different nanowires heights can be drawn via AGM measurements. This allows to remove the influence of different lengths since AGM measurements are done on small chunks of samples and the curves are normalized. The hysteresis loops for different average nanowires heights for high nickel content and high cobalt content nanowired membranes are shown on figures 4.15 and 4.16.

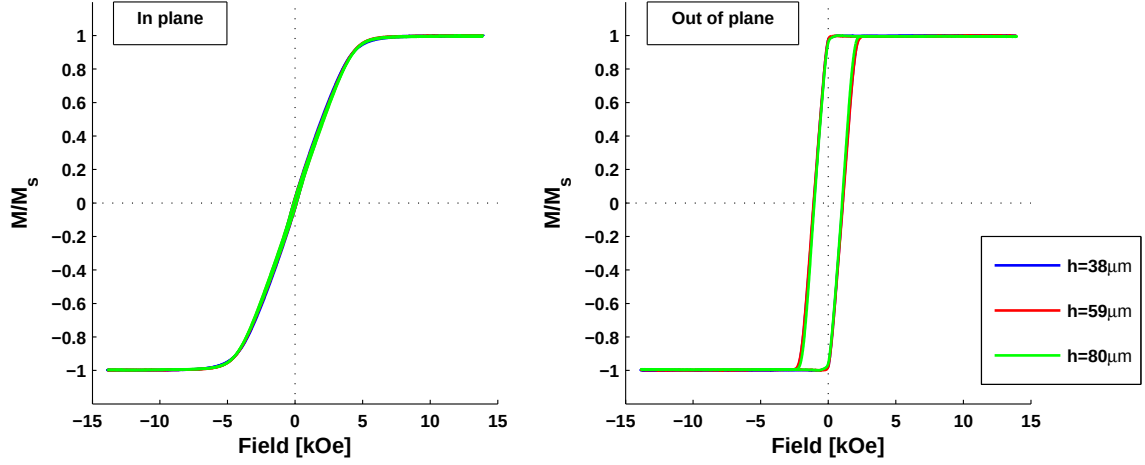


Figure 4.15: Hysteresis loops for SM containing high nickel content nanowires of heights $h = 38\mu m$, $h = 59\mu m$ and $h = 80\mu m$.

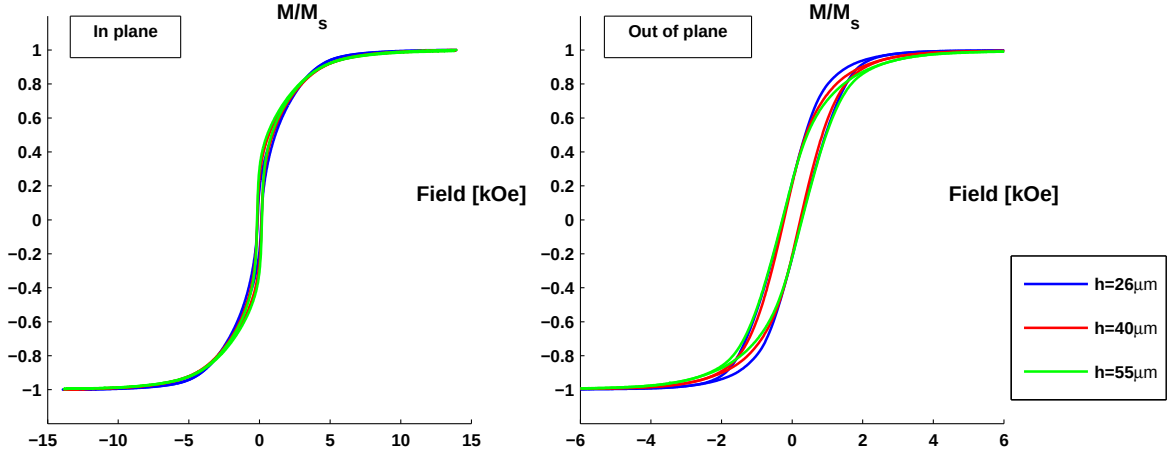


Figure 4.16: Hysteresis loops for SM containing high cobalt content nanowires of heights $h = 26\mu m$, $h = 40\mu m$ and $h = 55\mu m$.

We notice that almost no differences can be found for the different heights of nanowires grown in these experiments. The slight differences can be attributed to small changes in the measurements or deposition conditions. Hence, we can see that no extra anisotropies arise from the change in height of nanowires, which would lead to think that no significant magnetoelastic anisotropies are present in this kind of materials since magnetoelastic anisotropy would vary with the height and size of the nanowires. However, it has been found that the change in magnetoelastic contribution becomes negligible for heights around the ones considered here [43]. Hence, we would need to make the same comparison with even smaller nanowires in order to accurately discuss the presence or absence of magnetoelastic contribution.

4.7 Conclusion

In this chapter, two types of NiCo nanowires embedded in nanoporous anodized alumina membranes have been characterized and their filtering properties have been examined. It has been

shown that high nickel content NiCo nanowires behave like pure Ni while high cobalt content NiCo nanowires have a behaviour in between the pure Ni and the pure Co deposited from a solution of pH 5 which means that it has an important fraction of hcp crystals with magnetocrystalline anisotropy contribution perpendicular to the axis of the nanowires. The different types of membranes have shown negligible influence on the properties of the deposited nanowires.

The different measurements for the two types of NiCo nanowires allowed to extract some characteristic values of the deposited nanowires such as γ , the Landé factor and the anisotropy field in a membrane. The ideal single domain nature of the high nickel content nanowires only has also been shown.

The filtering properties have also been studied. First, the correct way to treat the transmission data has been discussed and some experiments have been proposed. The high nickel content NiCo nanowires have shown much better absorption than high cobalt content. It can be explained by the single magnetic domain nature of the high nickel content nanowires leading to a saturation magnetization of the whole sample at zero magnetic field if previously saturated. Furthermore, this type of nanowires show sharper peak suggesting a low Gilbert damping constant and a more homogeneous crystalline structure. It has been suggested that the magnetic field has no influence on the absorption other than the shift in resonance frequency but further experiments should be performed in order to confirm this result. The increase in power absorption with the length of the nanowired zone and the height of the nanowires has been demonstrated without being precise enough to allow to draw relations. However, the power absorption enhancement in dB seems to be linearly related to the length of the nanowired zone.

Finally, a power absorption of more than 99% has been shown using a high nickel content NiCo nanowired membrane at zero applied magnetic field suggesting that magnetic nanowire arrays could efficiently eliminate some frequencies of an electromagnetic signal. Since the measurements show smaller power absorption at higher applied fields, further investigation should be performed.

Chapter 5

Filtering SIW

The filtering of microwave signals is particularly interesting when the filtering material is embedded in a waveguide structure because signal is then guided through the filtering structure which minimizes the losses at other frequencies than the resonance frequency. Microstrip lines are too lossy for these applications while waveguides are commonly used.

We have described the substrate integrated waveguide (SIW) as an alternative to traditional waveguides. We have seen that rectangular waveguides and SIW are intrinsic high-pass filter since the electromagnetic waves with a frequency smaller than the cutoff frequency of a waveguide are not propagated. We have also seen that the operating bandwidth was then between the cutoff frequency and 2 times this same frequency. In order to filter out some unwanted frequencies, a filtering structure has to be introduced in the SIW. It is then interesting to use the magnetic nanowires as filtering materials and hence add a band-stop filter in addition to the high-pass filter. They can also be used to form the walls of the SIW and replace the vias traditionally used. The feasibility of a nanowires-based SIW has already been demonstrated [3].

The goal of this section is to optimize the fabrication process and compute the ideal dimensions of the SIW and the nanowires to efficiently filter out the electromagnetic (EM) signal around one frequency while maximizing the transmission at other frequencies. This can be done using the finite-element analysis based software, Comsol Multiphysics as well as through analytical developments. Since it is not possible to accurately represent the nanowires in the SIW on Comsol due to their small sizes, the zones composed of discrete ferromagnetic nanowires in an alumina substrate were represented as a continuous material having the averaged properties of the nanowires-charged substrate. Some experimental considerations will also be introduced.

5.1 Design

We want to use a nanoporous alumina membrane to build a filtering SIW. First of all, in order to have a SIW, 2 metallic plates and 2 rows of vias are needed. The copper layer usually deposited via electron beam evaporation on one side of the alumina membrane to act as the cathode for the electrodeposition is already one of the metallic planes. A second metal layer must hence be added on the other side of the membranes.

The traditional vias shown in the theoretical chapter can be replaced with overfilled metallic nanowires walls. Despite their very small diameter, the possibility to have multiple rows of nanowires should reduce the radiation losses.

The ferromagnetic nanowires used to filter the electromagnetic waves inside the SIW should be placed between the two walls of nanowires. They must not be overfilled (contrarily to the ones forming the walls) in order to allow good transmission through the waveguide at all frequencies in the operating bandwidth (except the resonance frequency). Overfilled nanowires on the whole width of the SIW would prevent any propagation through the SIW.

Finally, the SIW must be compatible with traditional transmission lines with 50Ω impedance. Therefore, a Deslandes geometry or a quarter-wave transformer should be added at the extremities of the waveguides and linked directly to a microstrip that can be connected to transmission lines.

Top view and sectional view schemes of the design are represented on figures 5.1, 5.2 and 5.3.

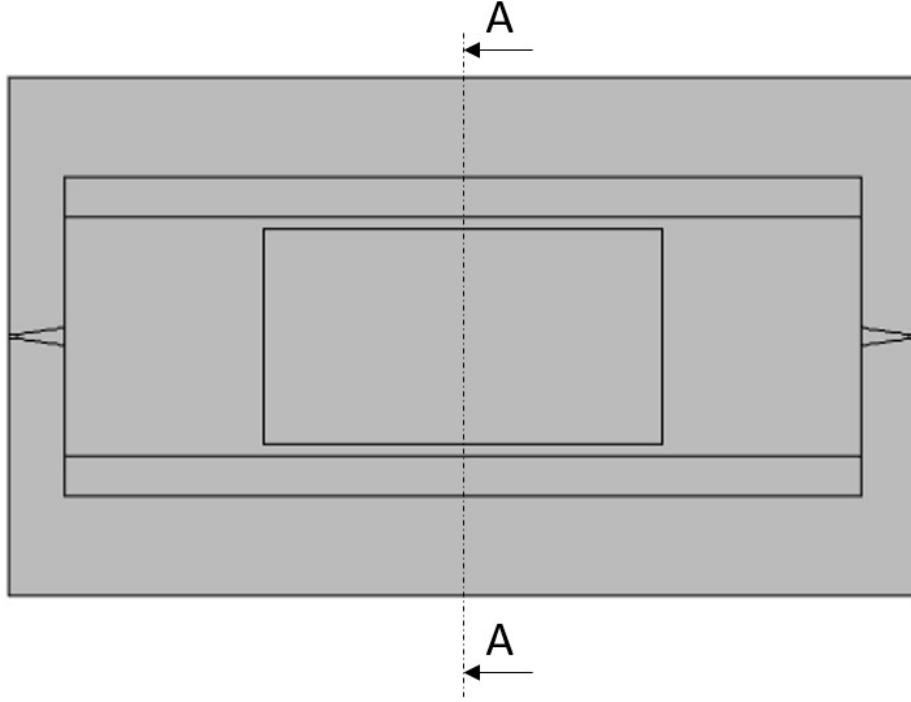


Figure 5.1: Top view scheme of a filtering SIW using nanowires adapted with the Deslandes geometry. The microstrips at the extremities are connected through trapezoids to the waveguide structure. The two rectangles on the sides are the walls formed of metallic nanowires (coated with a metallic plate and hence not visible in practice) and the rectangle at the center is formed of ferromagnetic nanowires (also coated with metallic plate and practically invisible). The sectional view A is visible on figure 5.3.

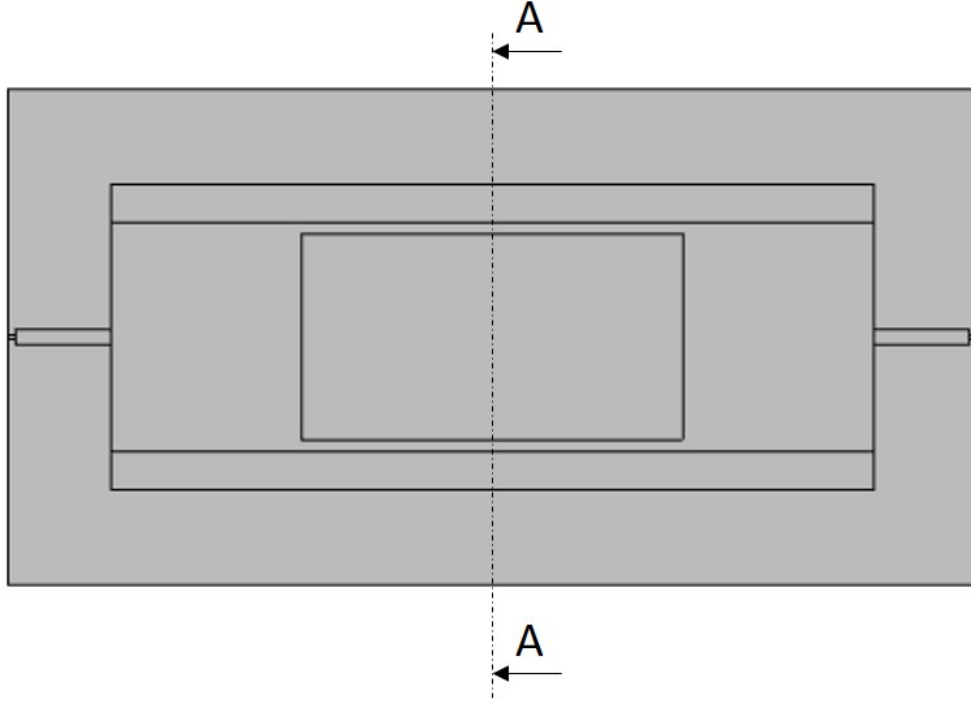


Figure 5.2: Top view scheme of a filtering SIW using nanowires adapted with a quarter-wave transformer. The microstrips at the extremities are connected through rectangles (quarter-wave transformers) to the waveguide structure. The two rectangles on the sides are the walls formed of metallic nanowires (coated with a metallic plate and hence not visible in practice) and the rectangle at the center is formed of ferromagnetic nanowires (also coated with metallic plate and practically invisible). The sectional view A is visible on figure 5.3.

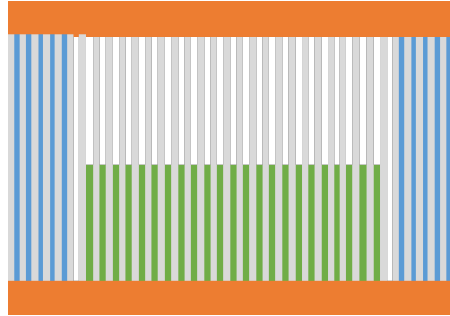


Figure 5.3: Sectional view scheme A of a SIW using metallic nanowires as walls (in blue) and ferromagnetic nanowires as filtering materials (in green) embedded in the nanoporous membrane (in grey) on which two metallic plates are deposited (in orange) represented on figures 5.1 and 5.1.

5.2 Fabrication

5.2.1 Initial process

The initial process was described by V. Van Kerckhoven in 2016 [3]. It consists in a series of electron beam evaporation, electrochemical deposition and picosecond laser patterning steps represented on figure 5.4.

The first step (a) is the deposition of the metallic layers: one copper metallic layer to serve as the cathode for electrodepositions and one aluminum layer.

The second step (b) is the laser patterning of the aluminum layer at the places we want to deposit nanowires. Note that for filtering applications, a third rectangle should be etched in the middle to allow the deposition of the filtering ferromagnetic nanowires.

The electrodeposition can then be performed in the patterned zones (c). The aluminum layer on top is used because of the bad electrodepositions on aluminum. Indeed, the nanowires grown for the walls need to overfill. If a copper layer was used, at the precise moment one nanowire would overfill, most of the subsequent deposition would go directly on the copper layer that would be directly linked to the cathode and hence become part of the cathode. On aluminum, thanks to the bad deposition, we can expect the unfinished nanowires to still grow.

The aluminum layer must then be removed (d) because the patterned zones need to be covered again with metal and coating again without first removing the aluminum layer would induce important variations in the final upper metallic layer.

A copper layer is again deposited on top of the membrane (e) to act as the last metallic wall of the SIW.

Finally, the copper metallic layer is patterned in order to allow connections with transmission lines.

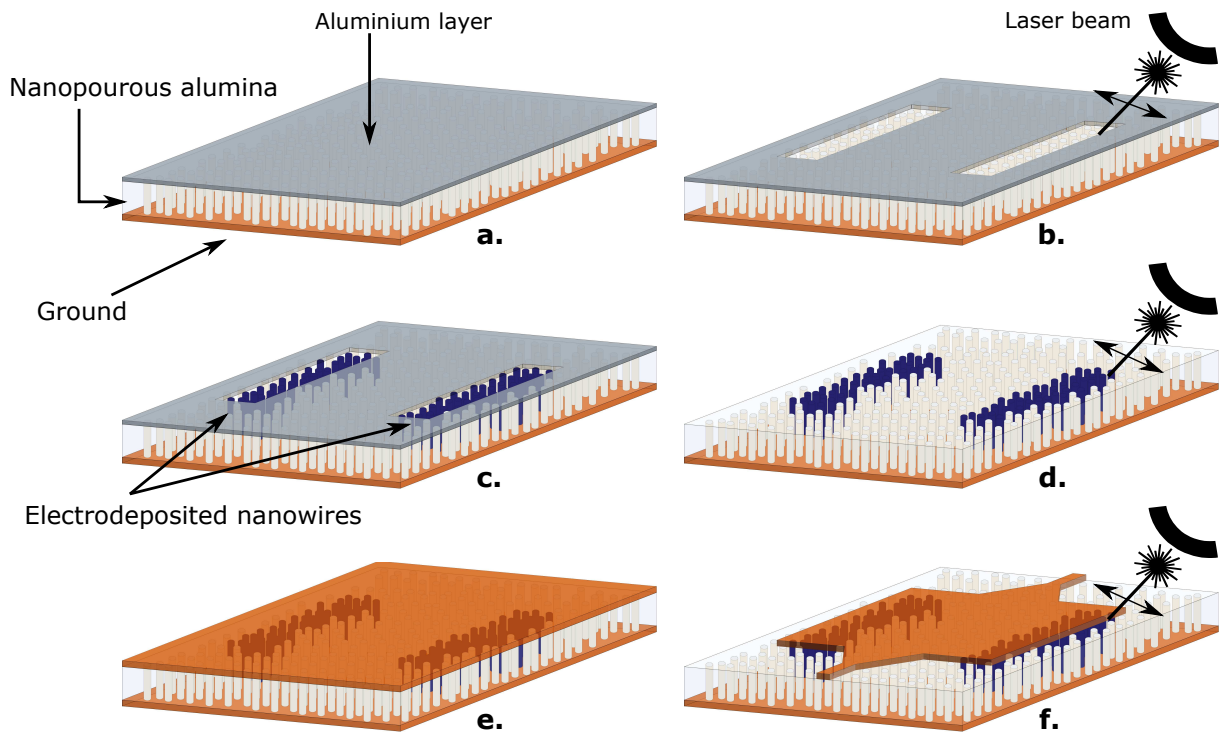


Figure 5.4: Description of laser patterning experimental process for a simple SIW. (a) Deposition of the top masking aluminium layer ($500nm$) and bottom copper cathode ($1\mu m$). (b) Laser etching of the desired pattern. (c) Nanowires growth by electroplating. (d) Complete removal of remaining aluminium with the laser. (e) New copper deposition on membrane top face. (f) Laser design of strip line and connections [3].

Some tests were performed on a membrane with deposited aluminum in order to determine the optimal laser parameters for etching. Different number of passes and laser power were tested in order to find an optimal etching process. The best quality of pores after etching was found for the laser etching 7 times the pattern at 200mm/s with a power of 0.11 W and a frequency of 40kHz.

5.2.2 Alternative process

The first limitation of the fabrication process presented here above is the difficulty to overfill the nanowires forming the walls. Indeed, despite the aluminum layer on top, when one nanowire overfills the membrane, the other ones will have difficulties to still grow and overfill. A possible solution was then to replace aluminum with a non-metallic layer like Poly(methyl methacrylate) (PMMA). It should however be checked that the laser can efficiently etch this material without attacking too much the membrane.

A second problem that arised was the unavailability of the picosecond laser due to the failure of one of the engines of the plateau. Hence, the three laser steps had to be eliminated. The two first laser steps could be eliminated by using a deposition cell with the desired pattern which would also solve the first problem described. This cell (represented on figure 5.5) allows the solution to access only the wanted locations on the membrane. The disadvantages of this cell are the unique size compared to the versatility of the laser process and the difficulty to isolate the zone between the two walls. The third laser step could also be replaced by a mask with the correct pattern put on the membrane during the last electron beam evaporation step (e). Again, the main limitation of this solution is the unique size available.

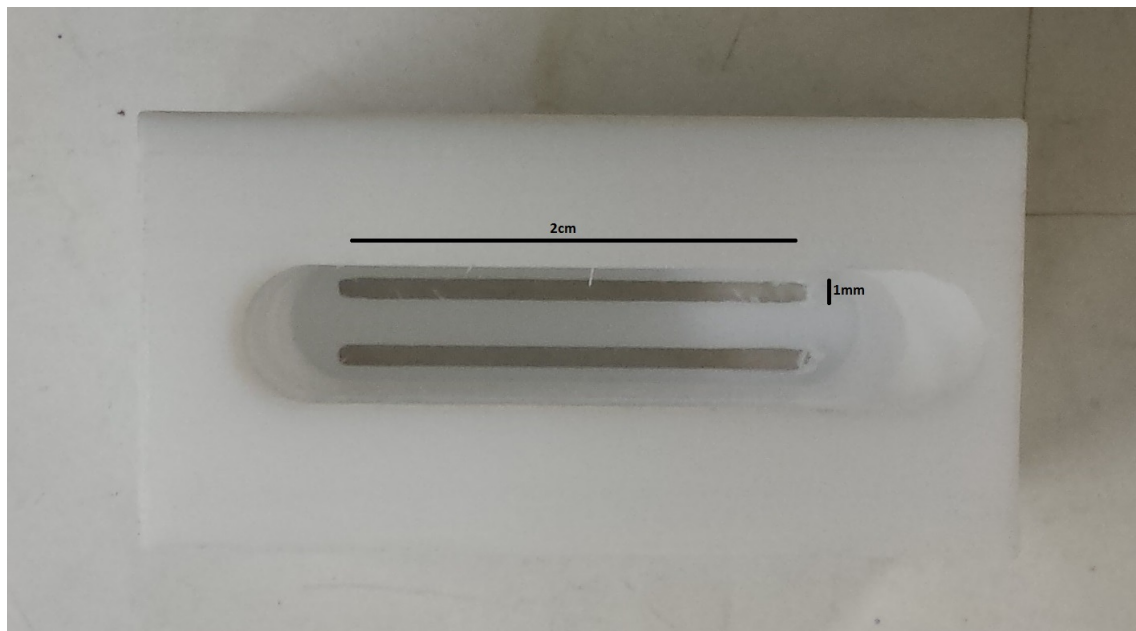


Figure 5.5: Picture of the electrodeposition cell with two holes to allow the deposition of the two nanowired walls of the SIW.

Note that apart from the cell with double holes, none of the solutions could be experimentally tested due to time constraints.

5.3 Optimization of the dimensions

In order to study the ferromagnetic resonance absorption of nanowires in a SIW, we need to optimize the dimensions considering the transmission of an electromagnetic signal and the range of frequencies we want to filter. This range will be determined by the ferromagnetic material chosen for the tests. We first wanted to work with CoFe for his high saturation magnetization and high resonance frequency but the very long deposition showing many irreproductibilities (see appendix A) motivated us to first work with NiFe that usually shows more predictable behaviour.

The different optimal dimensions have been found using analytical developments, numerical simulations with Comsol multiphysics and practical considerations. The final results are shown in table 5.1 for adaptation with both the quarter-wave transformer and the Deslandes geometry. Note that for the Deslandes geometry, the width of the adapter on the side of the microstrip line is the width of the microstrip line itself and is hence not shown in the table. The adapter width described is then the width of the other extremity.

	Quarter-wave transformer	Deslandes geometry
Microstrip line length	$200\mu m$	$200\mu m$
Microstrip line width	$105\mu m$	$105\mu m$
Waveguide length	$2cm$	$2cm$
Waveguide width	$6mm$	$6mm$
Walls width	$1mm$	$1mm$
Nanowired zone height	$50\mu m$	$50\mu m$
Nanowired zone width	$5.4mm$	$5.4mm$
Nanowired zone length	$2cm$	$2cm$
Adapter length	$2.175mm$	$2.4mm$
Adapter width	$400\mu m$	$700\mu m$

Table 5.1: Dimensions of a SIW with a zone of ferromagnetic NiFe nanowires.

The influence of size changes on the transmission and the absorption for the described dimensions will be shown with the quarter-wave transformer adapter since only the adapter length changes. The optimized structure will then be compared for both geometries.

5.3.1 Microstrip line dimensions for low transmission losses

The signal first penetrates the substrate in a microstrip line. The dimensions of this microstrip line must be such that it matches the standard 50Ω impedance of transmission lines. The characteristic impedance of a microstrip line Z_0 can be computed based on the relative permittivity of the substrate ϵ_r , the thickness of the substrate d and the width of the conductor deposited above the substrate W as shown in equation 2.112.

For a porosity p of 10% (SM), the relative permittivity of the nanoporous alumina membrane ϵ_r is defined by

$$\epsilon_r = \epsilon_{alumina} \cdot (1 - p) + \epsilon_{air} \cdot p \quad (5.1)$$

where $\epsilon_{alumina}$ the relative permittivity of pure alumina is 9.8 and ϵ_r the relative permittivity of air is 1. Hence, $\epsilon_r = 8.92$. We also know the height of the substrate ($100\mu m$) and we want Z_0 to be 50Ω . We then find the width needed for the conductor: $105\mu m$.

We can now take a look at the influence of the length of the microstrip line (c_{long}). Figure 5.6 shows that it has very few influence on the transmission except a slight decrease with the length of the microstrip line. The value of $200\mu m$ was kept for the other simulations because it is almost the smallest length that allows connection.

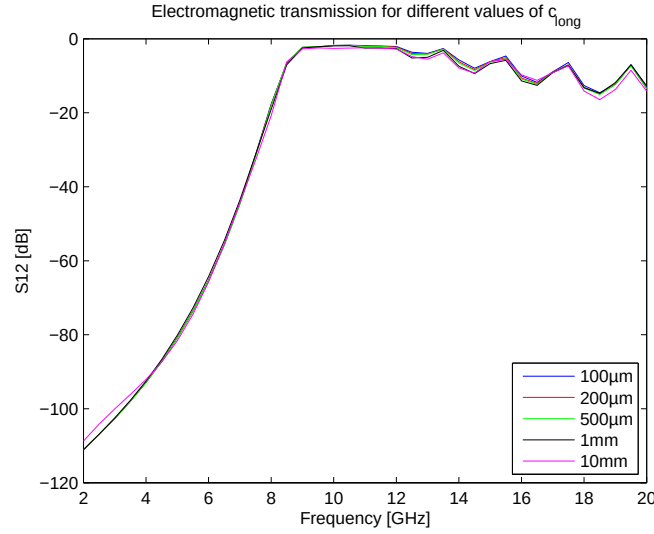


Figure 5.6: Electromagnetic transmission as a function of the frequency for different values of the microstrip line length ($100\mu m$, $200\mu m$, $500\mu m$, $1mm$, $10mm$).

5.3.2 Ideal waveguide length and width

For the waveguide to have an ideal size with respect to filtering applications, two criteria have to be taken into account :

- The FMR frequency has to be between the cutoff frequency of the dominant mode of the device TE_{10} (almost no propagation under this frequency) and the apparition of mode TE_{20} (more perturbations over this frequency).
- The FMR frequency has to be in a frequency zone matching the linear regime of the imaginary part of the propagation constant as a function of the frequency.

The cutoff frequencies of the dominant mode, i.e. TE_{10} and the second mode of transmission appearing TE_{20} (and hence overmoding the system) are:

$$f_{c10} = \frac{1}{2a\sqrt{\mu\epsilon}} \quad f_{c20} = \frac{1}{a\sqrt{\mu\epsilon}} \quad (5.2)$$

Since $\epsilon_0 = 8,85418782 \cdot 10^{-12} F/m$ and $\mu = 1 * \mu_0 = 4 \cdot \pi \cdot 10^{-7} H/m$ (for a dielectric), and since the substrate has a relative permittivity 8.92, we can get a graph of the cutting frequency for TE_{10} and TE_{20} as a function of the width of the waveguide. This is represented on figure 5.7 for the most common ferromagnetic materials.

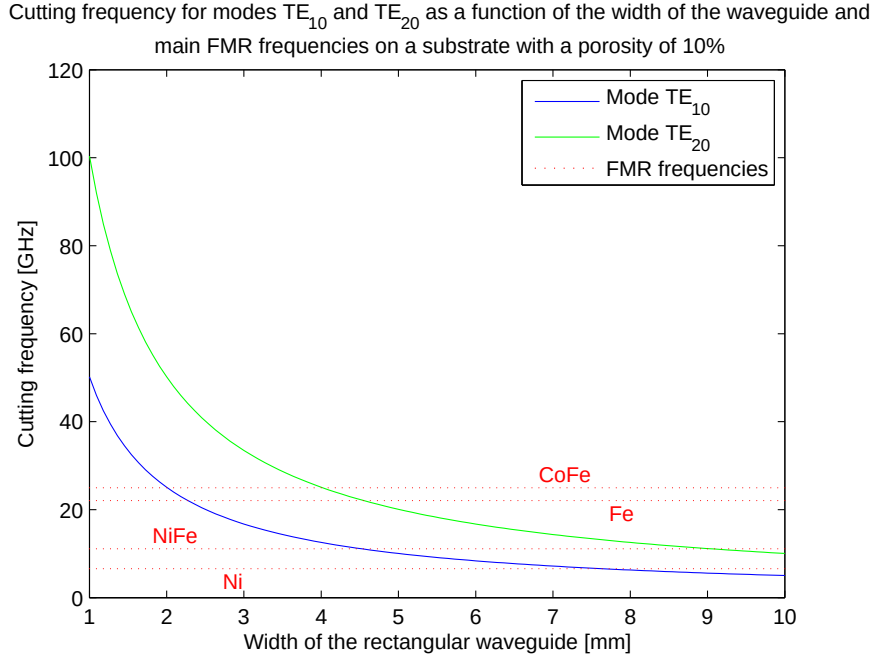


Figure 5.7: Plot of propagation modes in the waveguide TE_{10} and TE_{20} as a function of the width of the waveguide and the FMR frequencies of Ni, Fe, $Ni_{80}Fe_{20}$ and $Co_{55}Fe_{45}$ in a Smart membrane with a porosity of 10%.

Given the FMR of the main ferromagnetic materials, in order to have dimensions suitable for multiple materials, we should rather choose a width in the range between $2.5mm$ and $4mm$ for $Co_{55}Fe_{45}$ and Fe and in the range between $8mm$ and $9mm$ for $Ni_{80}Fe_{20}$ and Ni. For NiFe only, the suitable dimensions are between $4.5mm$ and $9mm$ approximately.

Now, we want to have a look at the location in the linear zone of the imaginary part of the propagation constant β . Since the phase velocity v_p of the electromagnetic waves propagating in the waveguide is described by

$$v_p = \frac{\omega}{\beta} \quad (5.3)$$

If β has a linear dependence on ω , v_p is a constant and hence the signal is not deformed during his propagation, the medium is nondispersive. It has been shown that in a lossless medium, if $\omega < \omega_{FMR}$, β is 0. For $\omega > \omega_{FMR}$, $\beta = \sqrt{\omega^2 \mu \epsilon - p^2}$ where $p = \omega_c \cdot \sqrt{\mu \epsilon}$. The resulting curve is plotted for an arbitrary width of the waveguide of $4mm$ for an alumina medium (see figure 5.8).

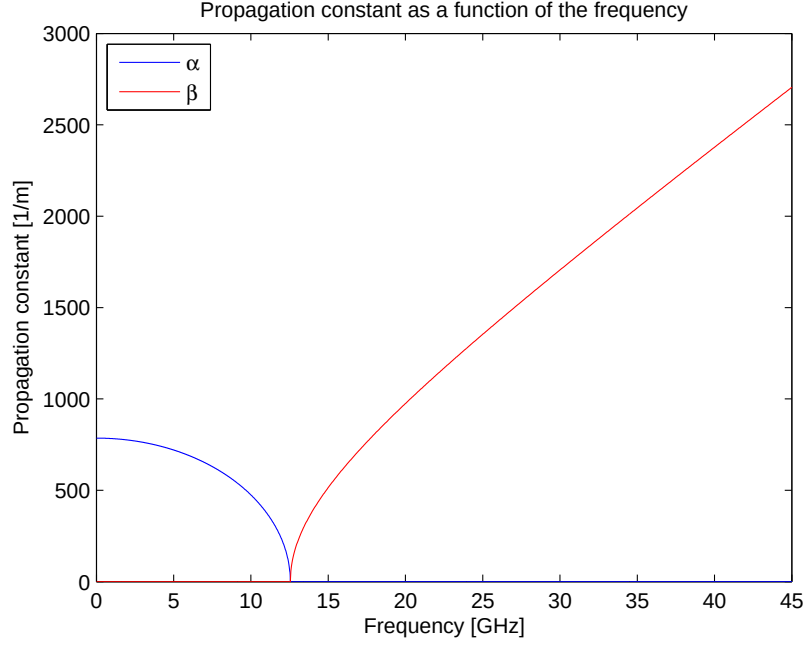


Figure 5.8: Real and imaginary part of the propagation constant as a function of the frequency in a SIW on SM with a width of $4mm$.

We can see that the linear regime is reached if we are not too close from the cutoff frequency (on the graph, we can assume linear regime around $5GHz$ over the cutoff frequency).

We can now precise the optimal width for NiFe only using Comsol Multiphysics. The transmission is represented with and without the nanowired zone in order to check both the general transmission and the precise position of the absorption peak. We can see on figures 5.9 that a $6mm$ large waveguide matches efficiently the absorption frequency of NiFe situated at $12GHz$ which is quite close to theoretical results ($11.1GHz$, see table A.2). This width also allows good transmission around the peak.

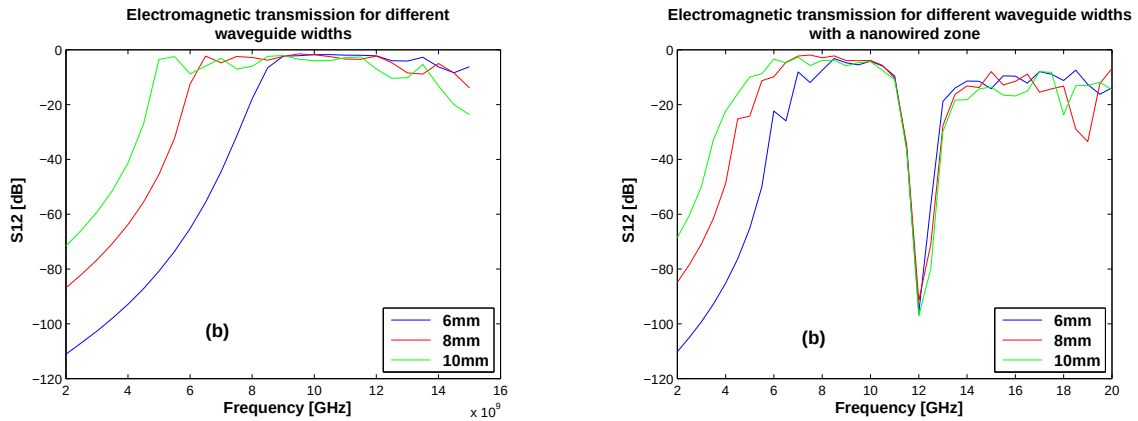


Figure 5.9: Electromagnetic transmission through the SIW for widths of $6mm$, $8mm$ and $10mm$ without any filtering nanowires (a) and with a nanowired zone (b).

Let us now check the influence of the length of the waveguide, even though we prefer a length around $2cm$ for experimental reasons (support size). We can see on figures 5.10 that the main

influence of the length of the waveguide is on the frequencies smaller than the cutting frequency. We have a sharper cutoff in the case of longer waveguides. We can also see on figure (b) that the transmission slightly decreases with the length of the waveguide. We conclude that even from a theoretical perspective, 2cm length is a good compromise between an efficient transmission and a sharp cutoff frequency.

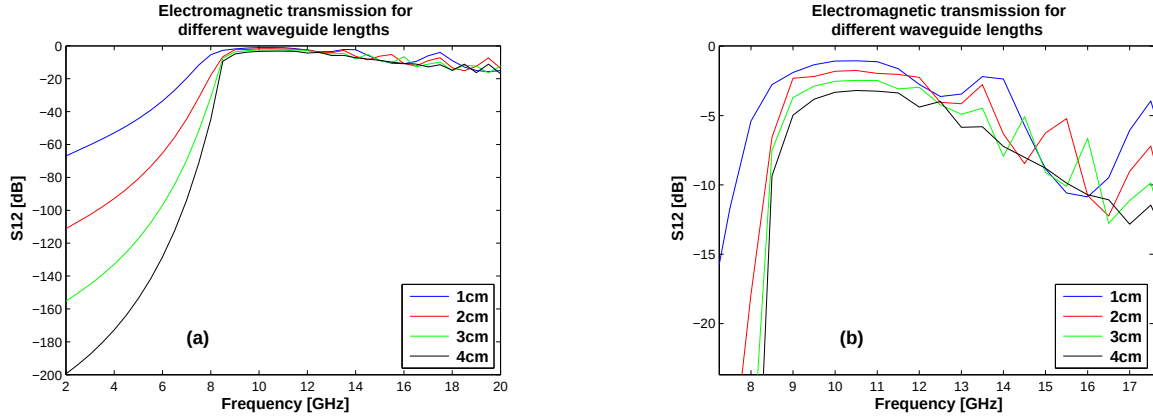


Figure 5.10: Electromagnetic transmission as a function of the frequency for waveguide lengths of 1cm , 2cm , 3cm and 4cm (a) and zoom on the curve on the range of interest (b).

5.3.3 SIW walls dimensions for good isolation

The length of the walls is logically the length of the waveguide and hence 2cm . In order to determine the minimum width, we have to consider the walls as an assembly of punctual nanowires shielding the electromagnetic waves. We can analytically describe the shielding behaviour using the concept of skin effect [17].

The skin effect describes the attenuation of an alternating current oscillating with a pulsation ω entering a conductor of conductance σ , permittivity ϵ and permeability μ . The characteristic skin depth in a good conductor ($\sigma > \omega\epsilon$) is given by δ :

$$\delta = \sqrt{\frac{2}{\omega\mu\sigma}} \quad (5.4)$$

In such a case, the electric field (and therefore the magnetic field) is decreasing exponentially with γd where d is the distance travelled through the conductor and γ is a complex number described as follows:

$$\gamma = (1 + i)/\delta \quad (5.5)$$

Using those equations, we can find the thickness of good conductor that the electromagnetic wave needs to cross in order to become negligible. This distance corresponds to a distance of a few times δ .

We must first find the values of the conductivity of the metal nanowires used for the walls (typically Ni or Cu). This can be done by using pseudo-four terminal sensing on nickel and copper nanowires. The resistance of the nanowires can then be measured. A descent of temperature is also needed in order to compute the Residual Resistivity Ratio (RRR) defined as

$$RRR = \frac{R_{290K}}{R_{20K}} = \frac{\rho_{290K}}{\rho_{20K}} = \frac{\rho_r + \rho_{i_{290K}}}{\rho_r} \quad (5.6)$$

where R is the resistance, ρ the resistivity, ρ_r the residual resistivity and ρ_i the ideal resistivity.

The curves showing the resistance as a function of temperature for nickel and copper are shown on figure 5.11. From this, we extract for Ni $R_{20K} = 7\Omega$ and $R_{290K} = 36.335\Omega$ and for Cu $R_{20K} = 316.5\Omega$ and $R_{290K} = 623\Omega$. These values are for a few nanowires connected together through their overfilled part. Then, it is possible to isolate ρ_r using the values of ρ_i available in [5] (for Ni $\rho_{i290K} = 7 \cdot 10^{-6}\Omega cm$ and for Cu $\rho_{i290K} = 1.7 \cdot 10^{-6}\Omega cm$). From this we find for Ni $\rho = \rho_r + \rho_{i290K} = 8.67\mu\Omega cm$ and for Cu $\rho = \rho_r + \rho_{i290K} = 3.45\mu\Omega cm$. We can then deduce the conductivities $\sigma = 1/\rho$: $\sigma_{Ni} = 1.15 \cdot 10^7 S/m$ and $\sigma_{Cu} = 2.89 \cdot 10^7 S/m$. Note that these values are not completely accurate since contact resistance are added in order to connect the heads of the nanowires together.

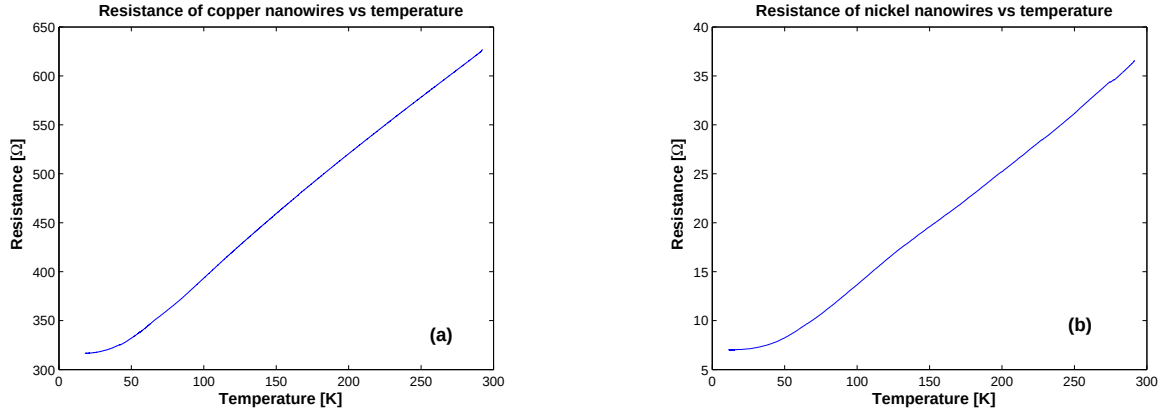


Figure 5.11: Resistance vs temperature curves for copper nanowires (a) and nickel nanowires (b).

In order to get an upper boundary of the skin depth, it is better to compute the result with copper since it has a magnetic permeability much smaller than Ni (relative permeability close to 1). Using also the value found for the conductivity ($\rho_{Cu} = 2.89 \cdot 10^7 S/m$) and the lowest value of the frequency of electromagnetic wave propagating in the SIW ($\sim 8GHz$), we get $\delta = 1.05\mu m$.

Since only the nanowires are good conductors, this is the total thickness of nanowires the signal has to cross. The fact that the signal doesn't travel all that distance at once does not theoretically change anything since the same attenuation is obtained by crossing n times a distance d of good conductors or by crossing a distance nd at once, or in other words, because $(e^{-\gamma d/\delta})^n = e^{-\gamma nd/\delta}$. Since the porosity of the alumina is around 10%, when the EM wave crosses a distance x of the walls, it will cross a distance $x\sqrt{0.1} = 0.316x$ of nanowires. The width of the walls should therefore be around $3.31\mu m$ to efficiently confine the EM signal.

This result gives us a good approximation of the minimum size of the walls and clearly shows that the smallest technically feasible walls are large enough to efficiently achieve their function. Even if we consider that globally only 10% of the nanowires will effectively overfill and if we neglect the others, the minimum wall size is still $10.46\mu m$ which is smaller than the minimum achievable sizes of the order of $1mm$. Hence, this value of $1mm$ will be kept.

5.3.4 Nanowired zone height, length and width for sharp and deep absorption

Now that the geometry of the waveguide has been discussed, let's consider the optimization of the nanowired zone. In order to absorb the EM signal, a zone of nanowires placed inside the

waveguide is used. This zone is composed of ferromagnetic nanowires with a height smaller than the height of the substrate. As we have seen before, nanowires connecting the bottom and top electrode would largely decrease the transmission at all frequencies. We can now discuss the optimal dimensions of this zone of nanowires.

Let's first have a look at the optimal width of the zone. The length is set at one half of the total length of the waveguide (2cm) and the height is set at one half of the height of the substrate ($100\mu\text{m}$). We can see on figure 5.12 that the larger the zone, the better the absorption which seems logical since the nanowires can intercept more of the EM signal if the plot is larger. The absorption does not increase linearly with the width. It can be understood since specific polarization of the waves will be obtained at specific places in the waveguide leading to different absorptions. Note that the biggest size considered is 5.4mm and not 6mm (width of the waveguide). Indeed, in order to grow different sizes of nanowires in the central zone and the walls with sometimes different materials, the plot and the walls must be physically separated. This separation needs to be in the order of at least a few hundred micrometers such that it is technically possible to achieve precise depositions.

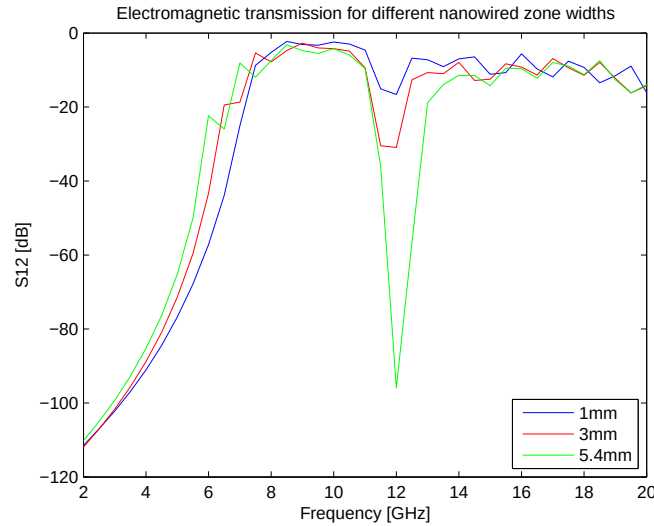


Figure 5.12: Electromagnetic transmission as a function of the frequency for nanowired zones of 1mm , 3mm and 5.4mm (with a height of $50\mu\text{m}$ and a length of 1cm) in a SIW of 2cm length, 6mm width and $100\mu\text{m}$ height.

Now we can look at the optimal length of the nanowired zone. Again, we expect to have a better absorption with a longer zone but we also want to optimize the transmission. It is expected that very long nanowired zone could deteriorate the transmission. We can see on figure 5.13 that the absorption indeed increases with the length of the zone more or less linearly. However, we cannot clearly determine the influence of the length on the transmission. The optimal length of the nanowired zone could therefore be the whole length of the waveguide but this should be tested further experimentally.

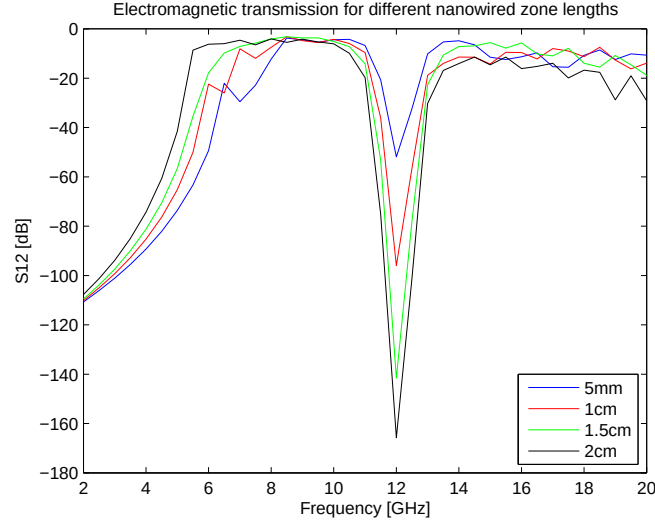


Figure 5.13: Electromagnetic transmission as a function of the frequency for different nanowired zone length ($5mm$, $1cm$, $1.5cm$, $2cm$) in a SIW of $2cm$ length, $6mm$ width and $100\mu m$ height.

Finally, the nanowires height must also be discussed. The plan is again to compromise between a good absorption (high nanowires) and a good transmission (low nanowires). The transmission for different heights is given on figure 5.14. We see that, as expected, the absorption increases with the height of the nanowires. This increase seems to be linear with the height until $50\mu m$ filling but not for heights over this value. However, this time we observe a quick decay of the transmission with the height. Furthermore, the linewidth of the peak also increases with the height of the nanowires. A good compromise is obtained for a height around 50% of the substrate ($50\mu m$). We also observe a shift in frequency. Since it hasn't been observed experimentally in the previous chapter, we can assume that this shift is induced by some numerical errors in Comsol.

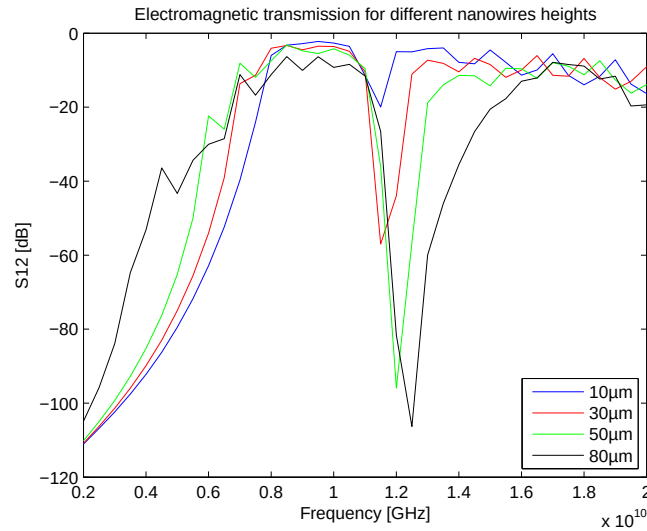


Figure 5.14: Electromagnetic transmission as a function of the frequency for different nanowires heights ($10\mu m$, $30\mu m$, $50\mu m$, $80\mu m$) in a SIW of $2cm$ length, $6mm$ width and $100\mu m$ height.

Note that the input voltage used for the simulations didn't have any influence on the resulting curves. This is an argument in favor of the use of the relative absorption discussed in the previous chapter.

5.3.5 Quarter-wave transformer dimensions for impedance match

In order to efficiently (with minimum losses) transmit the signal from the microstrip to the waveguide, matching the impedances is required. A simple and practical circuit to achieve that is the quarter-wave transformer presented in the theoretical chapter. As indicated by its name, it has to be a length corresponding to a multiple of a quarter of the wavelength going through it. While we are not going to work with only one frequency and wavelength, we are mostly interested by the frequencies around the absorption peak of NiFe. Comsol simulations showed that the peak was situated on 12GHz and extended to 10GHz and 14GHz . We are interested in maximizing the transmission at frequencies around the peak of absorption (between the cutoff frequency and 10GHz or between 14GHz and the apparition of mode TE_{20}). Let's optimize the transmission at 14GHz . The wavelength in the microstrip λ can be computed using the speed of light in vacuum $c = 3 \cdot 10^8\text{m/s}$, the considered frequency $f = 12 \cdot 10^9\text{Hz}$ and the effective permittivity given by equation 2.111:

$$\lambda = \frac{c}{f\sqrt{\epsilon_e}} = 8.7\text{mm} \quad (5.7)$$

Hence, the length of the quarter-wave transformer should be 2.175mm . This ensures a good impedance matching at 14GHz but this matching will not be as good for other frequencies.

The optimal width of the quarter-wave transformer t_{larg} has to be determined numerically. The optimal value found for the width with figure 5.15 is $400\mu\text{m}$. Other values decrease the quality of the transmission.

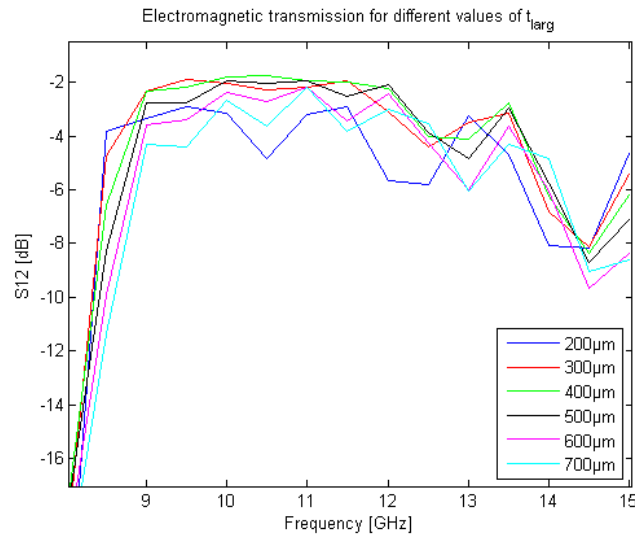


Figure 5.15: Electromagnetic transmission as a function of the frequencies for different width of the quarter-wave transformer ($200\mu\text{m}$, $300\mu\text{m}$, $400\mu\text{m}$, $500\mu\text{m}$, $600\mu\text{m}$, $700\mu\text{m}$).

5.3.6 Comparison with Deslandes geometry

The optimization of the trapezoid used in the Deslandes geometry gives a size of the adapter of $700\mu\text{m}$ width on the side of the SIW (the width on the other side being the width of the microstrip line i.e. $105\mu\text{m}$) and 2.4mm length.

The transmissions for the two optimized impedance matching structures are compared on figure 5.16. We see that the quarter-wave transformer shows slightly better transmission at low frequencies and worse transmission at high frequencies. Globally, the Deslandes geometry allows to work with a larger operating bandwidth than the quarter-wave transformer.

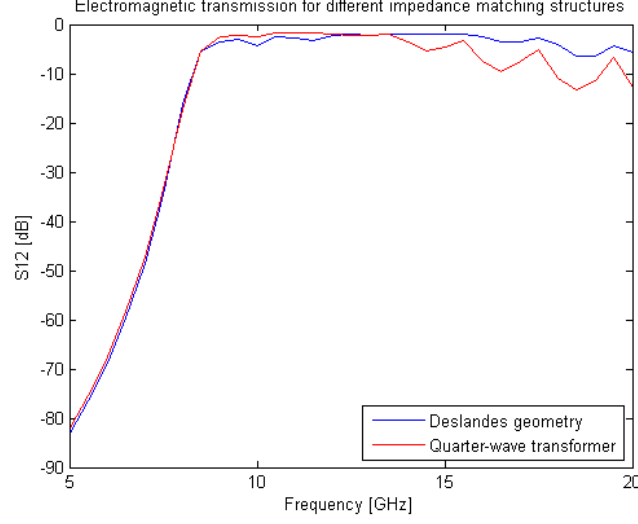


Figure 5.16: Electromagnetic transmission vs frequency of the optimized device with a Deslandes adapter and a quarter-wave transformer.

5.4 Conclusion

The fabrication process of a SIW with ferromagnetic filtering NiFe nanowires has been discussed. Alternatives to the process initially proposed have been suggested and the first experimental preparations and equipment fabrication have been done.

The optimal dimensions have also been described using analytical and numerical descriptions. The whole geometry of a filtering SIW using NiFe nanowires as filtering materials have been theoretically optimized.

Due to the laser breakdown, neither the alternative process nor the optimized geometry could be fully tested. Hence, some tests should now be performed in order to witness the suitability of the alternative process and the effective transmission obtained for an experimental device with the described dimensions. It would also be interesting to observe experimentally the influence of the nanowired zone on the transmission and on the absorption because of its critical impact. Furthermore, since the nanowires cannot be precisely represented in Comsol, some differences could be observed with the numerical results.

Finally, the impact of the geometry used should also be checked since finite-element analysis are always less precise on edges and corners that are typically numerous at the extremities of the impedance matching structure. Hence, the conclusion favoring the Deslandes geometry should also be validated.

Chapter 6

Experimental issues and key observations

During the different experiments performed, a few different issues have been encountered. These led to some observations and sometimes some solutions. The main issues that will be presented hereafter concern particularly the electrochemical deposition process but also cover some mechanical issues as well as the already mentioned laser breakdown.

6.1 Irreproducibility of the electrodepositions

One major issue faced during many experiments was the irreproducibility of the electrochemical depositions (ECD). Some results presented in appendix A already show that the electrochemical depositions of almost all materials don't have the same behaviour from one time to the next and that the evolution of the deposition time with for example the porosity is not predictable. This phenomenon can be associated to the important height of the nanowires deposited (minimum a few tens of microns). Small changes in the nanoporous membrane, the solution, the mounting of the electrodeposition cell or the cathode can lead to important differences in the deposition process. This was investigated a step further during the experiments with NiCo.

The two first tests of electrodeposition of high nickel content nanowires were done on the same Sy (but on different spots). The current vs time curves of those two electrodepositions on a membrane we will call "Sy1" are represented on figure 6.1.

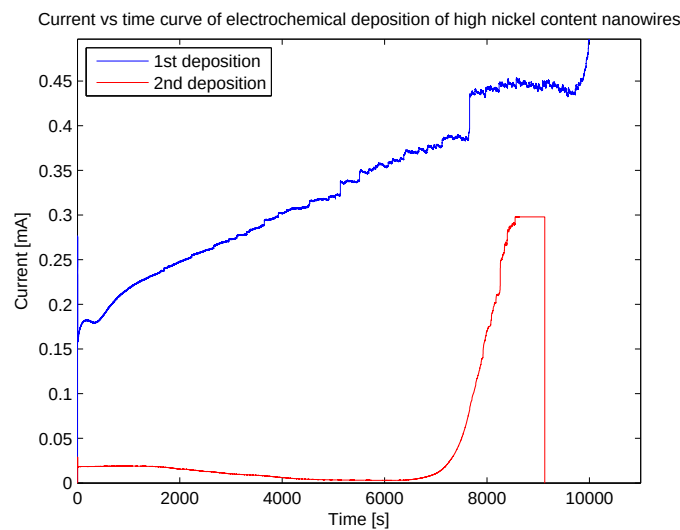


Figure 6.1: Current-time curves of two consecutive depositions of high nickel content NiCo nanowires on the same membrane Sy1.

A cleaning of the membrane was executed between the two steps with deionized and demineralized water, followed by a drying. However, it is very clear that the deposition is completely different on the second deposition. While the overfilling seems to happen in both cases between 7000 and 8000 seconds (overfilling is less clear for the first deposition), the total charge deposited before overfilling for the second ECD is at least one order of magnitude smaller than the one for the first ECD. This is an indication that the number of grown nanowires is much smaller than during the first deposition despite the fact that the same zone was immersed in electrolytic solution. Based on the experience of regular users of ECD in the labs of UCL, it seems that such difference occurs quite often when depositing multiple times on the same membrane, even if the spots deposited are not in contact. It is hard to precisely identify why this happens, because the two zones are physically separated. However, the acidity of the solution may deteriorate the membrane around the deposition zone and the conductivity of the cathode probably changes after each deposition. Constraints may also appear with the apparition of nanowires, leading to physical changes in the membrane. It may also come from the rubber used to prevent leakage during deposition. Since it is pressed on the membrane around the deposited zone, some dirt can stay and clog pores or destroy them under the pressure.

On a second membrane "Sy2", high cobalt content NiCo nanowires were deposited on 4 different zones. This time, the influence of cleaning with ultra pure (distillated) water was investigated. After the first deposition, a cleaning of 2 hours into ultra pure water was done. Then a classic cleaning with deionized and demineralized water was performed before the third deposition. Before the fourth deposition, another cleaning of 30 minutes in ultra pure water was done. Note that the fourth deposition was stopped before the overfilling for practical reasons. One more zone of the same composition was deposited on "Sy1" after a very long cleaning in ultra pure water (2 days). This deposition is the fourth on the membrane Sy1. The current vs time curves of the five high cobalt content NiCo nanowires ECD are shown in figure 6.2.

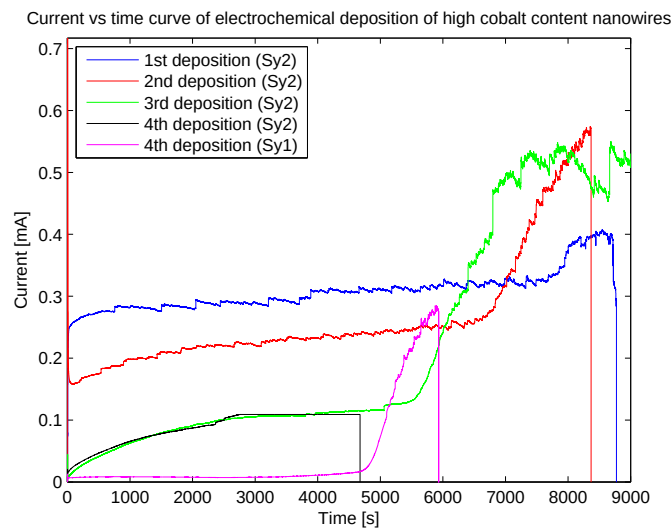


Figure 6.2: Current-time curves of four consecutive depositions of high cobalt content NiCo nanowires on the same membrane Sy2 and one on Sy1 (fourth zone deposited on this membrane). Cleaning with ultra pure water was done for 2 hours between first and second deposition and for 30 minutes between third and fourth deposition. Classic cleaning was done between second and third deposition. A 2 days cleaning in ultra pure water was also done on Sy1 before the deposition.

Here, we can see that, while the two first depositions do not have identical ECD curves, they seem

quite similar compared to the two curves shown in figure 6.1. This tends to show some efficiency of the cleaning with ultra pure water. The third deposition done after classical cleaning already shows much weaker current, which is coherent with previous observations. The fourth deposition done after 30 minutes cleaning with ultra pure water is almost identical to the third one. Hence, we can see that the cleaning with ultra pure water seems to limit or completely prevent the decrease of deposition current (and probably thus the number of deposited nanowires) without being able to invert it. It then seems that the membrane deteriorates a bit more after each consecutive deposition which would lead in each case to the observed decrease in the measured current during ECD after each consecutive deposition.

The last deposition of the same material on Sy1 shows that even very long cleaning with ultra pure water isn't enough to efficiently "repare" the membrane. This confirms that the damages on the membrane due to previous depositions cannot be canceled with a cleaning. It also confirms the tendency of the overfilling time to decrease with each consecutive deposition.

It is quite clear that multiple depositions on the same membrane will never give very comparable results in term of current of deposition and total charge deposited. Hence, the overfilling for the first deposition on a membrane should be the most reliable. Therefore, in order to obtain a certain percentage of filling of the pores, the deposited charge was taken as this percentage of the charge at the beginning of the overfilling of the first depositions on the test membranes.

The ECD shown before were done on membranes only used for the purpose of testing the overfilling time and charge specific to high nickel content and high cobalt content NiCo nanowires in Sy. The following membranes were used to measure the FMR with a microstrip line and hence didn't have to be overfilled by nanowires. The size of the deposited nanowire zones varied in order to observe the change in filtering of the electromagnetic waves. Hence, we can now compare the ECD curves for the same material deposited on different clean Sy but only by normalizing the current with the deposition area. Indeed, the deposition current is supposed to scale more or less linearly with the surface since the number of pores and hence nanowires scales linearly with the surface. However, we can't expect the scaling to be purely linear due to other effects like ion migration that can vary with greater sizes. The charge per surface of high nickel content nanowires was taken as 90% of the charge of the first test on Sy1 just before overfilling while the charge of high cobalt content nanowires was taken as 68% of the charge of the first test before overfilling on Sy2. The different sizes of the deposition zones were 3.9mm for the test then 3.9mm, 5.9mm, 10mm for high nickel content NiCo nanowires and 3.7mm for the test then 3.9mm, 5.9mm, 10mm for high cobalt content NiCo nanowires. The current-time curves for the two different materials are represented on figure6.3.

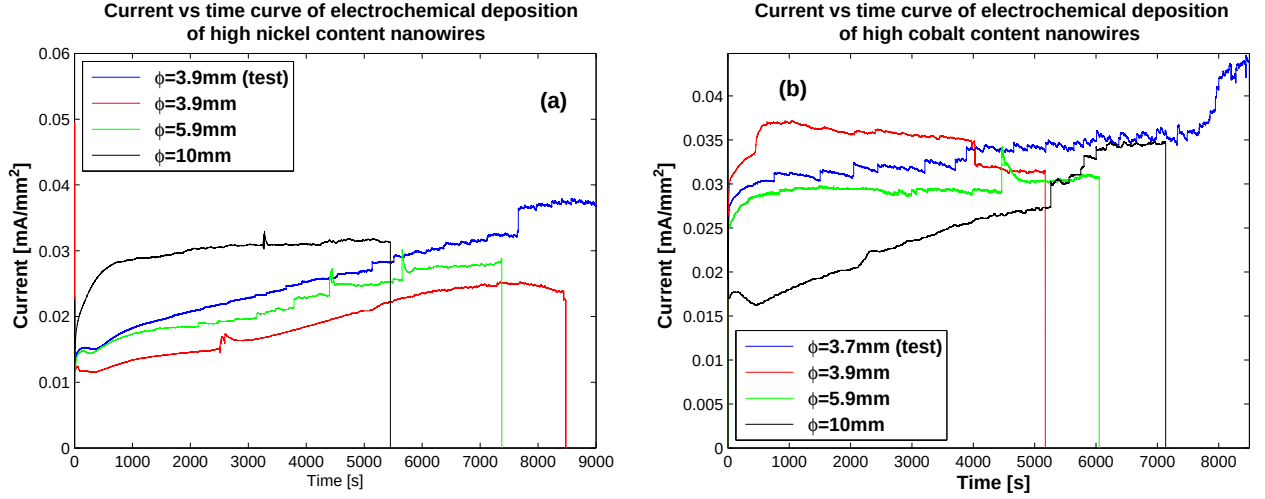


Figure 6.3: Current-time curves of high nickel content NiCo nanowires (a) and high cobalt content NiCo nanowires (b) deposition on different Sy done on zones of 3.9mm of diameter (test membrane), 3.9mm , 5.9mm and 10mm (a) and 3.7mm of diameter (test membrane), 3.9mm , 5.9mm and 10mm (b).

First of all, let's note that the curves are much more comparable than consecutive depositions on the same surface which again proves that multiple depositions on the same sample intrinsically change the system and therefore the deposition process. The normed currents are more similar for lower nickel concentration, especially if we look after longer deposition time. This is quite surprising since pure nickel depositions are usually very stable and reproducible and hence we would expect high nickel content NiCo to behave the same way. Let's also note that the depositions on larger zones deviate more from the others. It seems quite logical since the anode can't be anymore at a similar distance from all pores unlike for small deposition zones.

Even with those curves much more similar than for consecutive depositions on the same membrane, the difference is far from negligible. Ideally, the current per surface should be similar for all depositions, leading to similar deposition times since the same charge per surface is deposited. However, we can see that the difference in deposition time varies by several thousand seconds. The sectional views of the resulting nanowired membranes are shown in appendix B.1. Linking the current-time curves and the height deposited, we can see that higher normalized deposition currents are associated to smaller nanowires heights. It seems logical since higher currents are linked to more nanowires growing. Hence, for the same deposited charge, the nanowires can grow less, leading to smaller nanowires. This phenomenon is probably caused by difference of qualities of the membranes, difference in anodes, cathodes or placements of these, or difference in cleanliness of the membranes. This leads to important differences that can lead to some difficulties, especially when a specific height of the nanowires is required. We therefore decided to use the overfilling time rather than the overfilling charge for subsequent experiments.

In order to make longer deposition zones, SM were needed. New tests on those membranes were therefore required. Multiple depositions of nanowires with high nickel content or high cobalt content were again done on different SM. The current-time deposition curves of the same zone (3.9mm diameter) of these materials on different test membranes are shown on Figures 6.4.

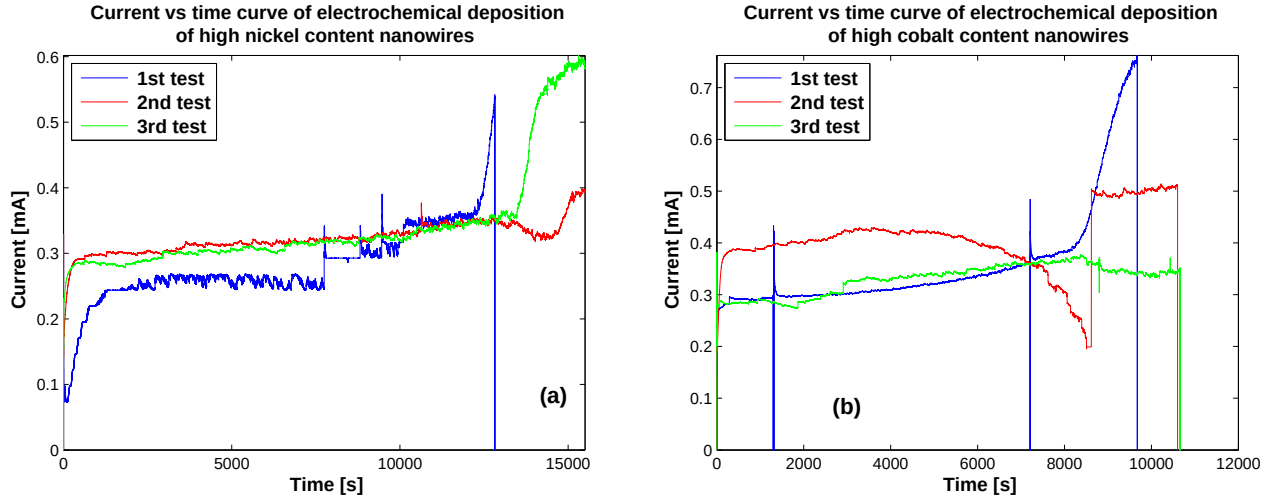


Figure 6.4: Current-time curves of high nickel content nanowires (a) and high cobalt content nanowires (b) deposited on zones of 3.9mm of diameter on different SM membranes.

We can see that, for the high nickel content, the deposition current of the first test is quite different from the deposition currents of the second and third tests that are quite similar. This can result from the presence of bubbles on the deposition zone during the ECD, a difference in membrane quality or differences in anodes and cathodes for example. The current is lower than for the other tests yet the overfilling happens sooner. While the deposition current for the two other tests is similar, the overfilling occurs with around 1330s difference.

For the high cobalt content, only the first test was performed until the end. Indeed, the second test didn't go well and the third test was not over even after several thousand seconds more than the first test and with a similar deposition current. Hence, for practical reasons, it had to be stopped and only the first test was considered reliable.

Given the high variability in deposited charge previously seen, the time was used as reference rather than the charge. For high nickel content, the overfilling time was taken as an average of the different deposition times, that is 13000s . For the high cobalt concentration, the overfilling time of the only complete test was taken, that is 8300s .

The same problem as before follows this irreproducibility of the deposition: the height of the nanowires is hard if not impossible to precisely control. The goal for the SM subsequently deposited was to reach heights of 30%, 50%, 70% and 90% on different samples for both high nickel and high cobalt content. The effective average fillings were 38%, 59%, 80% and 80% for high nickel content and 26%, 40%, 55% and 56% for high cobalt content NiCo nanowires. The results are not really close from the desired value, but the desired ratio in height are still better than on Sy as described before.

An example of this problem is illustrated on Figure 6.5. The goal here was to deposit high nickel content nanowires in a SM membranes with a height of $70\mu\text{m}$ (70% of the height). The deposition was therefore done during 9100s . However, after this time, the membrane was overfilled at the extremity close to the anode. Since the shortest time for overfilling during the tests was after 12000s , it was highly unlikely to have an overfilling so soon. And the fact that the anode was close to this point shouldn't change anything since the deposition zones during the tests were small enough to admit that the anode was close to every point on the surface of the membranes. But this overfilling still occurred, showing how problematic this non-reproducibility can be.

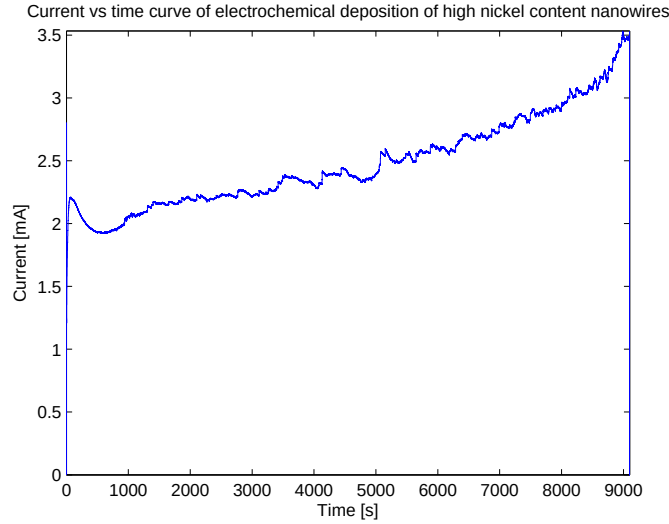


Figure 6.5: Current-time curve of the deposition of high nickel content NiCo nanowires on a SM. The deposition was supposed to fill 70% of the height of the membrane with nanowires.

6.2 Irregular nanowires height

The last result presented in the previous section also demonstrates the influence of the position of the anode: the zones closer to the anode have higher rates of deposition. Indeed, the nanowires have grown faster on the zone of the membrane close to the anode. This can be explained by a greater intensity of the electric field between the anode and the cathode leading to a greater number of ions being reduced at the bottom of the pores.

In fact, the differences in filling along the length of the SM have been observed in all samples. We talk here specifically about SM since they were much longer and hence the change in height was much more significant. It was already mentioned in chapter 4 that the heights given for the SM fillings were in reality an average of the heights of the nanowires at three points of the membrane (see pictures B.9, B.10 and B.11). Differences of filling up to more than 25% were indeed shown.

Hence, in order to obtain the more regular filling possible, the anode should be at the same distance from all points of the cathode. This is in practice not exactly feasible, but we could tend to this by placing the anode further from the surface with its projection in the middle of the deposition zone. Note that this would probably lead to slower deposition rate due to the increase in distance between the anode and the cathode. Another solution would be to use larger anodes.

6.3 Sources of membrane breaking

During the different experiments performed, a lot of samples broke. This is the biggest source of loss of samples and it was due to the weak mechanical resistance of anodized alumina membranes.

A few typical sources of breaking (and hence manipulations for which particular attention should be paid) can be identified. First, the pressure of the deposition cell (represented on figure 6.6) led to some breaking. It is however important to apply pressure in order to prevent leakages of electrolytic solution.

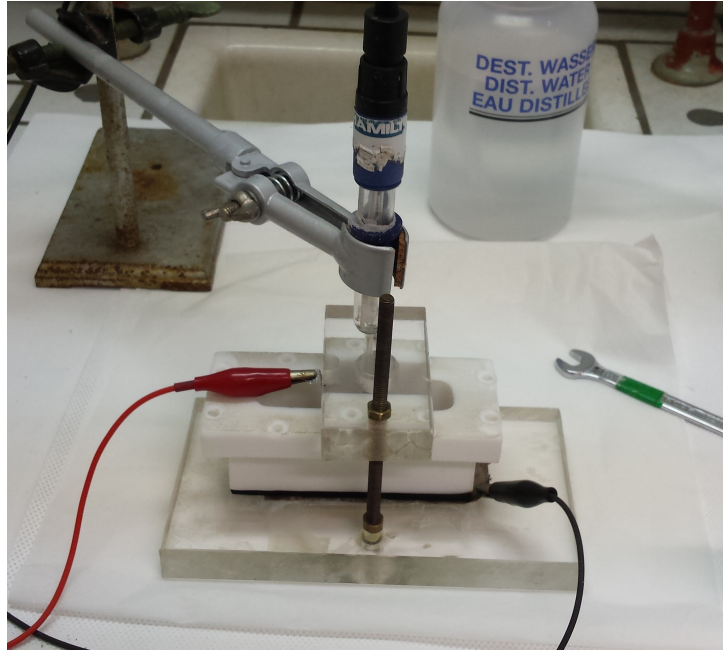


Figure 6.6: Picture of the typical electrodeposition cell. The black wire connects a metallic plane in contact with the cathode of the sample. A black rubber is used to prevent solution leakage. The white cell contains the electrolytic solution. The red wire maintains the anode in the solution. The reference electrode above is immersed in the solution.

Regular sticking and unsticking of adhesive paper on supports, for the electron beam evaporation, for the laser,... also led to a few breaking. Some drying caused the membrane to break when the air flux was too strong. Simple manipulations with the tweezers also led to failures of the membrane. Finally, the manipulation of the suspended membrane for VNA measurements (shown in figure 6.7) was also difficult to achieve.

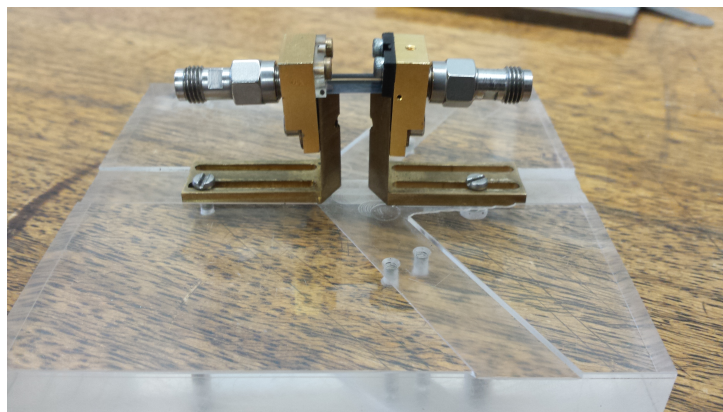


Figure 6.7: Picture of a 1cm long membrane suspended on the support for VNA measurements.

Globally, the Sy showed to be much more resistant than SM despite the similar heights, porosities and pore sizes.

6.4 Picosecond laser breakdown

The breakdown of an engine of the plateau of the picosecond laser led to the impossibility of fabricating SIW with nanowires. Furthermore, the SM (provided as large square of more than 10cm side) were too brittle to be cut by hand. Some cutting tests were also done in the Makilab using the 40W laser available there. However, this laser being classical (not picosecond or femtosecond), it couldn't cut the membrane otherwise than by burning it. This could have been enough to obtain an usable zone (with many material losses) but this introduced a lot of constraints leading to failures. Hence, the only solution to cut the SM was to use industrial picosecond lasers in Optec.

6.5 Conclusion and useful tips

From the data shown in this section, a few conclusions can be drawn. First, SM and Sy both show irreproductibility for the deposition of NiCo even if the way this irreproductibility appears is not exactly identical. Given the important height of the membranes ($100\mu m$), it is not surprising that a lot of different small perturbations (cleanliness, conductivity of the anode and cathode, position of these or difference of membrane quality) can lead to non-negligible differences in the deposited nanowires. We showed that it was then more precise to use the overfilling time rather than the overfilling charge in order to predict the filling of the membranes.

Secondly, it was shown that consecutive depositions on the same membrane will probably always lead to big irregularities. Ultra pure cleaning showed some efficiency but the quality of the deposition still decreased at each consecutive try. Thus, membranes should always be cut in pieces with the desired dimensions before any deposition. Cleaning of the membranes should also be performed before each deposition with at least ultra pure water. More advanced cleaning would further increase the homogeneity of the depositions on different membranes.

Third, since the device used (cell, cathode, anode) potentially has an influence of the deposition, the same device should always be used for experiments on the same subject in order to eliminate it as potential cause of variations in the depositions.

The influence of the anode position was also demonstrated. If small anodes compared to the deposition zone are used, they should then be carefully placed in order to have a similar distance from all points of the deposition surface.

The different sources of breaking were then presented, showing the particularly delicate steps and the superiority of the Sy when it comes to mechanical resistance.

Finally, the importance of the picosecond laser for the precise cutting of SM membranes was briefly discussed, showing that it is not easily replaceable.

Chapter 7

Conclusion and perspectives

This work has focused on the filtering properties of ferromagnetic nanowire arrays embedded in nanoporous alumina membranes. This study has been done through the characterization and comparison of two kinds of NiCo nanowires with different proportions of nickel and cobalt and through the optimization of a substrate integrated waveguide.

The magnetism was first generally described and the focus was then set on ferromagnetic properties like the hysteresis curves and the anisotropies and in particular the ferromagnetic resonance since it is the phenomenon used to filter electromagnetic signals. These phenomena were also described for the particular case of magnetic nanowires. Then, the electromagnetic theory of the waveguide was developed in order to understand the propagation of electromagnetic waves in this particular structure. The case of the substrate integrated waveguide was introduced as well as the microstrip lines since these are the two particular structures mentioned in this work to propagate electromagnetic waves.

In a second part, the experimental methods used to fabricate the nanoporous alumina membrane and to grow the nanowires were presented. The different instruments and techniques used to characterize and fabricate the samples were then described.

The magnetic characterization of the high nickel content and high cobalt content NiCo nanowires embedded in a nanoporous alumina membrane showed the presence of strong anisotropies. Concurrent anisotropy contributions parallel and perpendicular to the axis of the nanowires were identified for the high cobalt content nanowires. On the other hand, high nickel content nanowires showed strong uniaxial anisotropy. This last kind proved to be better adapted for filtering applications. Two reasons can be identified to explain this. Firstly, high nickel content nanowires showed almost ideal single magnetic domain nature leading to saturation magnetization for each nanowire at zero applied field. When the sample has been previously saturated with a strong magnetic field, it then keeps its magnetization until zero applied field. Secondly, it is likely that different crystalline structures and a high Gilbert damping constant in high cobalt content NiCo nanowires also led to smaller absorption and larger peaks.

In order to efficiently filter specific frequencies while maximizing the transmission at other frequencies, the absorption peaks should be sharp and deep. Ideal materials for this kind of applications should then behave as single magnetic domains when in the shape of nanowires, have a low Gilbert damping constant [45], large magnetic losses, comparable permittivity and permeability (to decrease the reflection losses) [46] and single crystal nanowires [47].

The influences of the magnetic field, the length of the nanowired zone and the height of the nanowires were also investigated. A first discussion was to determine the way the curves should be analyzed: using absorption relative to the transmitted signal without the ferromagnetic materials influence or using absolute values of the absorption. Some experiments were proposed to answer this question and it was decided to work with relative absorption. Some experimental results suggested that absolute absorption should be used while theoretical results with Comsol implied

the relevance of the relative absorption.

Due to large differences in nanowires heights and deposited zone lengths, it was hard to draw precise relations for the length and height influences of the nanowired zone. However, the increase of absorption with the height and the length of the deposited zone has been demonstrated. The absorption has shown to scale more or less linearly with the length of the deposited zone. The influences of both nanowires height and nanowired zone length should be investigated further with more sample of similar heights (to observe the influence of the length of the nanowired zone) or similar nanowired lengths (to observe the influence of the height of the nanowires).

It was shown for high nickel content NiCo nanowires that an absorption of more than 99% of the electromagnetic waves at one frequency was possible with nanowires of $80\mu m$ height deposited on a zone $2cm$ long. This result clearly shows the efficiency of some materials to eliminate certain frequencies of an electromagnetic signal.

The fabrication process of filtering substrate integrated waveguides using metallic nanowires as vias and ferromagnetic nanowires as filtering materials presented in [3] was then discussed. Some alternatives were proposed, and in particular the use of a PMMA layer used during one of the fabrication steps instead of an aluminum layer. The possibility to efficiently etch it with the picosecond laser should be demonstrated. The dimensions were then theoretically optimized for NiFe ferromagnetic nanowires as filtering materials. A device with the computed dimensions should be built in order to measure the experimental transmission in the SIW. The influence of the dimensions of the filtering zone should also be investigated experimentally in order to validate the numerical results.

Finally, some experimental tips were extracted from the difficulties encountered during the work. It was shown that for high membranes and nanowires (several tens of microns), it was more precise to observe the deposition time rather than the deposition charge to obtain specific heights. The positive influence of cleaning with ultra pure water was then shown. The importance of the position of the anode to obtain regular pore filling, especially for long deposited zone was discussed. The different source of breaking were also discussed and it was found that Sy were more mechanically resistant. Finally, the importance of the picosecond laser for precise cutting was developed.

Overall, this works shows the efficiency of nanowires embedded in nanoporous alumina membrane for the filtering of electromagnetic waves without any applied magnetic field. It also allowed to define the numerous characteristics needed for ferromagnetic nanowires to efficiently filter out some frequencies of an electromagnetic signal propagating in a structure like a SIW while optimizing the transmission at other frequencies. The experimental difficulties prevented to obtain an effective device (SIW) and to draw precise relations between the filtering capabilities and the dimensions of the nanowired zones but the general influences were clearly demonstrated. Hence, it should be part of further work to draw precise relations, find the most suitable material for filtering applications and show a finalized device using a SIW structure and ferromagnetic nanowires with strong sharp absorption at a specific frequency and good transmission at other frequencies in the operating bandwidth.

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Appendices

Appendix A

First tests on anodized alumina membranes fabricated in UCL

The first experiments were performed on alumina membranes double anodized in the labs of UCL ($50\mu m$ height, pore density around 30% and pores of diameters between 40 and $50nm$). The first experiments performed on these membranes had two main goals:

- Identify the needed enlarging time of the pores
- Make a first observation of the ferromagnetic resonance absorption of different materials and observe the associated frequencies

The different steps of the process are presented here below:

- After the double anodization was completed, the remaining aluminum layer was kept in order to be used as cathode.
- H_3PO_4 was poured on the membrane and let for 35 minutes. It is used to enlarge the pores.
- A mask with 25 holes of $12mm$ diameter (5 columns and 5 rows) was then attached on the membrane.
- H_3PO_4 was poured again in all the holes except the 5 from the first column and let for 5 min.
- H_3PO_4 was poured again in all the holes except the 10 from the two first columns and let for 5 min.
- This process was repeated obstructing the columns one by one in order to obtain holes with different time of exposition to H_3PO_4 in each column.
- Ni nanowires were deposited in each hole of the second row.
- NiFe nanowires were deposited in each hole of the third row.
- CoFe nanowires were deposited in each hole in the fourth row.
- Cu or CoFe nanowires were deposited in the holes of the fifth row.

The time chosen for enlargement is higher than $30min$ because it is known from experience that electrodeposition doesn't work very well with smaller enlargement time. Large enlargement times also weaken the membrane by enlarging the pores too much which is why the time is only increased of a few tens of minutes.

The charge of nanowires deposited was arbitrary fixed at $1C$ for each hole. The solutions and electric potentials used for the depositions are described in table A.1.

	H ₃ BO ₃ [g/l]	NiSO ₄ [g/l]	CoSO ₄ [g/l]	FeSO ₄ [g/l]	CuSO ₄ [g/l]	pH	E [V]
Ni	20	262.84	-	-	-	3.8	-1.1
NiFe	24.73	131.42	-	5.56	-	3	-1
CoFe	30	-	80	40	-	3.8	-0.9
Cu	30	-	-	-	212.23	3.8	-0.5

Table A.1: Electrolytic solutions based on NiSO₄, 6H₂O, CoSO₄, 7H₂O, FeSO₄, 7H₂O, CuSO₄, 5H₂O and H₃BO₃. The concentration of the different compounds in the solutions, pH of the solutions and potentials E° used for the electrodeposition of each solution are also given. The NiFe deposited from the described solution has 80% of Ni and 20% of Fe. The CoFe deposited from the described solution has 55% of Co and 45% of Fe [7].

We quickly realized that the deposition of copper nanowires was not good at all: the current curves for the deposition weren't similar to the other ones (short time and large current increase), and visual observation of the electrodeposited zones showed bad deposition. It is probably due to a poor quality of the solution. Indeed, when trying to adjust the pH of the solution with NaOH, some precipitation appeared and the solution was less clean than it is supposed to be. This is why the last few holes were filled with CoFe with a lower potential than in the fourth row in order to observe the differences ($-1.5V$ instead of $-0.9V$).

The electrodeposition time for the different materials were:

- Ni: between 800s (55' of enlargement) and 1300s (45' of enlargement)
- NiFe: between 1899s (55' of enlargement) and 2980s (35' of enlargement)
- Cu: 333s (35' enlargement) and 245s (40' enlargement)
- CoFe at $-0.9V$: between 4400s (35' enlargement) and more than 7000s (40' of enlargement)
- CoFe at $-1.5V$: quite constant times around 240s

This already shows the quite important irreproductibility of the electrodeposition process. The time needed for the deposition highly varies and this evolution can not even be related to the evolution of the enlargement times.

A scheme of the final membrane and its contents is shown on figure A.1.

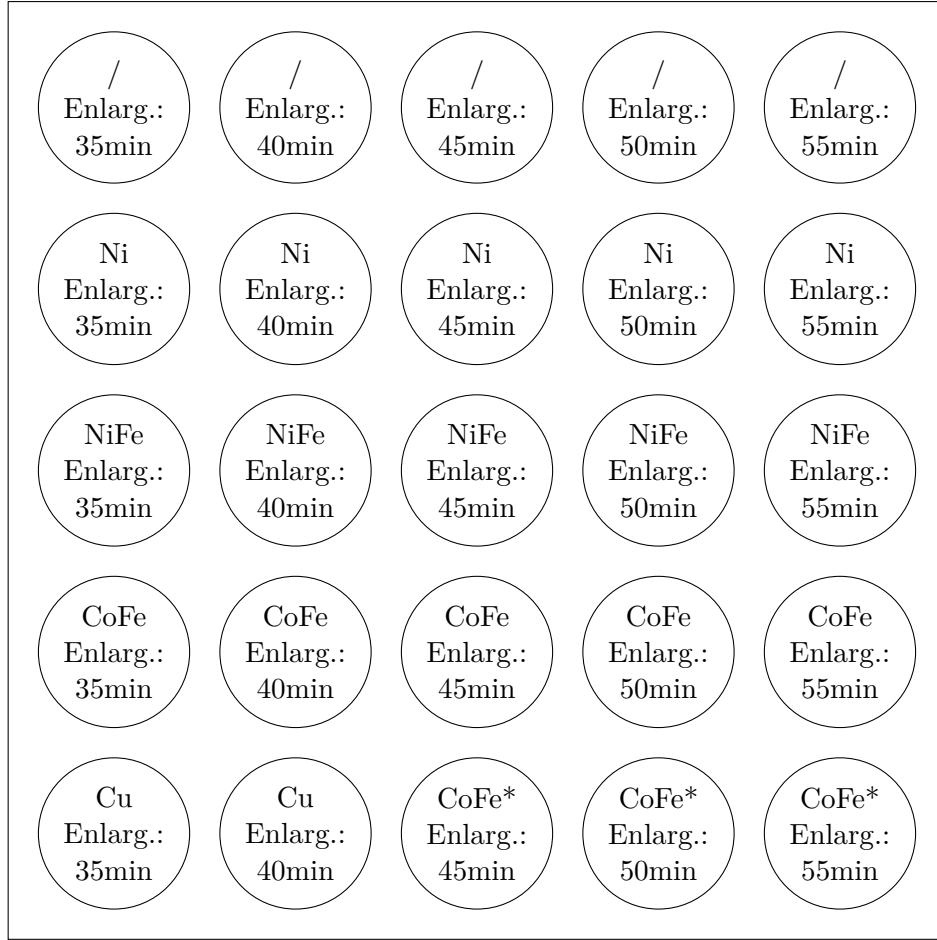


Figure A.1: Scheme of the membrane with the content of the nanowires and the enlargement time of the pores (CoFe* refers to the CoFe deposited with lower potential).

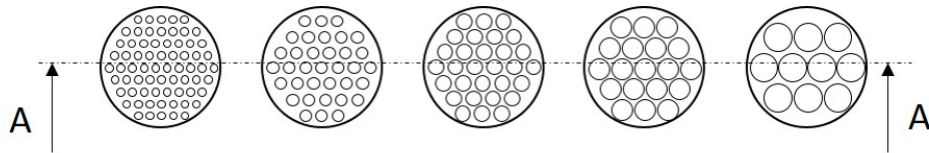


Figure A.2: Scheme of the top view of one row of deposition zones seen in A.1 showing the differences in porosity and pore sizes between the different zones (not representative of the real sizes). Sectional view A is shown in figure A.3.

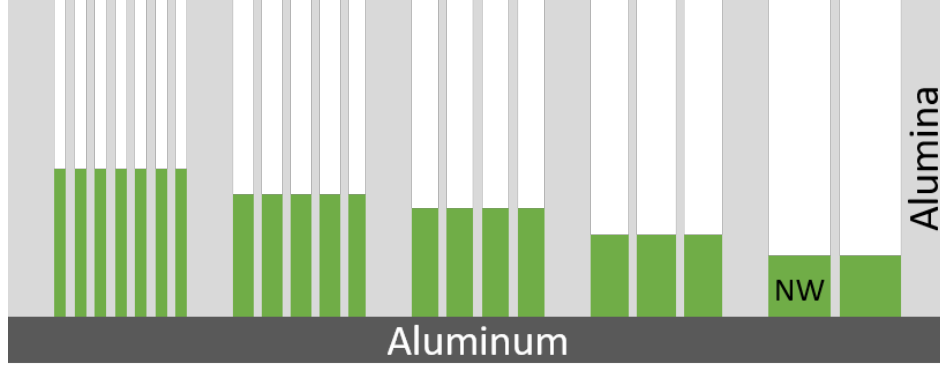


Figure A.3: Scheme of the sectional view of one row of deposition zones seen in A.1 showing the differences in porosity, pore sizes and nanowires heights between the different zones with the aluminum cathode in dark grey, the alumina in light grey and the nanowires in green (not representative of the real sizes).

Since all depositions happened in a similar way, we can conclude that an enlargement of the pores done by 35min of immersion in H_3PO_4 is already enough for the nanowires to grow. In order to attest the quality of this deposition and to make a first observation of the ferromagnetic resonance, the use of the VNA was required. Since it allows to measure the transmission of electromagnetic waves, it also allows to observe absorption by the medium, which is the effect of the ferromagnetic resonance. To make this measurements possible, microstrip lines had to be deposited. It was done using electron beam evaporation (5nm of Cr for the adhesion, 1.5 μm of Cu and 100nm of Au in order to maximize the electrical contact) without any mask, followed by picosecond laser etching in order to only let the desired width of the microstrip lines. The microstrip line width used was 200 μm in order to make the contact process easier.

It is possible to first theoretically compute the values of the ferromagnetic resonance frequency f_{FMR} at zero field using the equation 2.55 since we consider the case of nanowires with dominant magnetostatic anisotropy and negligible other sources of anisotropy:

$$f_{FMR} = \frac{1}{2\pi}(\omega_L + 0.5\omega_M(1 - 3p)) \quad (A.1)$$

with $\omega_L = \gamma\mu_0 H_0$ (and is thus 0 when no external magnetic field is applied) and $\omega_M = \gamma\mu_0 M_s$. Let us now apply this formula for the ferromagnetic materials used in this experiment, i.e. Ni, Fe, Ni₈₀Fe₂₀ and Co₅₅Fe₄₅. The corresponding Landé factors are given in A.2. Using the formula given in equation 2.27 with the correct values of the Landé factor g , the gyromagnetic ratios for those materials have been computed as well. Finally, using values of M_s found in the literature, the corresponding ferromagnetic resonance frequencies have been computed for a porosity $p = 0.3$ and are shown in A.2.

Material	g-factor	M_s [A/m]	γ [GHz/T]	f_{FMR} [GHz] (p=0.12)	f_{FMR} [GHz] (p=0.3)	f_{FMR} [GHz] (p=0.1)
Ni	2.2	$485 * 10^3$	193.46	6	0.94	6.57
Fe	2.088	$1715 * 10^3$	183.61	20.15	3.15	22.04
Ni ₈₀ Fe ₂₀	2.122	$850 * 10^3$	186.6	10.15	1.59	11.1
Co ₅₅ Fe ₄₅	2.115	$1917 * 10^3$	185.98	22.82	3.57	24.96

Table A.2: g-factor, gyromagnetic ratios, saturation magnetization and resonant frequencies on double anodized alumina membranes of different porosities for different common ferromagnetic materials [6, 10].

The measurements using the VNA were performed on different membranes using the constant magnetic field mode where a sweep in frequency is performed while the magnetic field is kept constant. The sample is always first magnetically saturated. The perturbations of transmission due to the mismatch are eliminated or at least reduced by subtracting the transmission curve at high applied magnetic field to the one at low magnetic field. The results are shown in figures A.4, A.5, A.6 and A.7. Note that there are less curves than deposited samples. It is the consequence of a few membrane breaks and some bad or unclear results due to unstable contacts or low quality depositions.

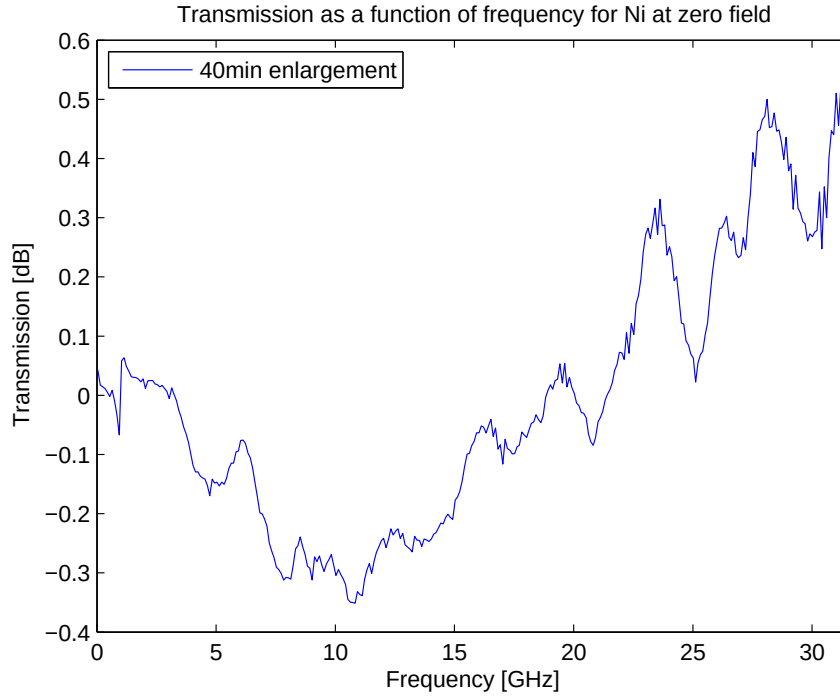


Figure A.4: Transmission vs frequency curve at zero magnetic field for nickel nanowires in an anodized alumina membrane made in UCL with 40min of pore enlargement time.

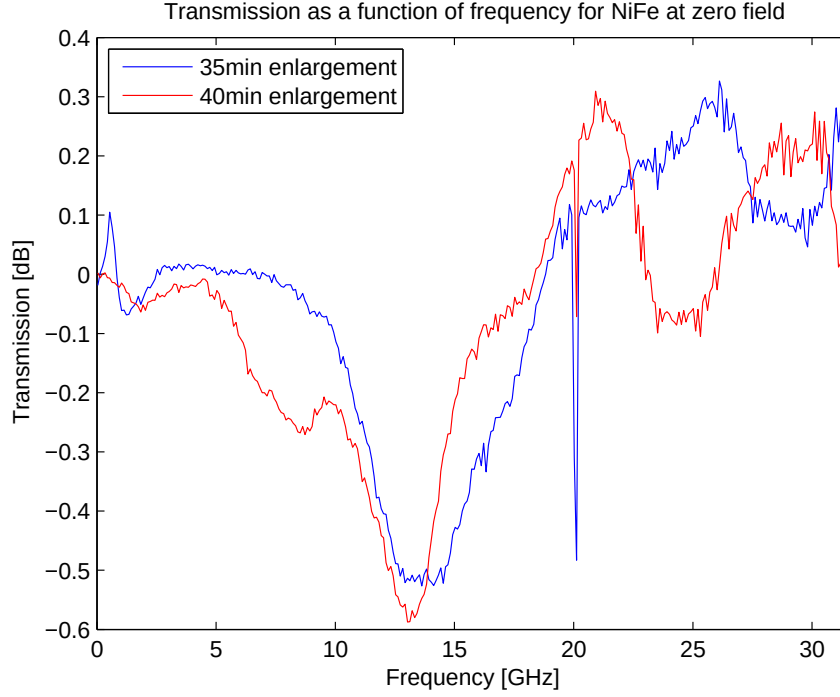


Figure A.5: Transmission vs frequency curve at zero magnetic field for nickel(80%)-iron(20%) nanowires in an anodized alumina membrane made in UCL with 35min and 40min of pore enlargement time.

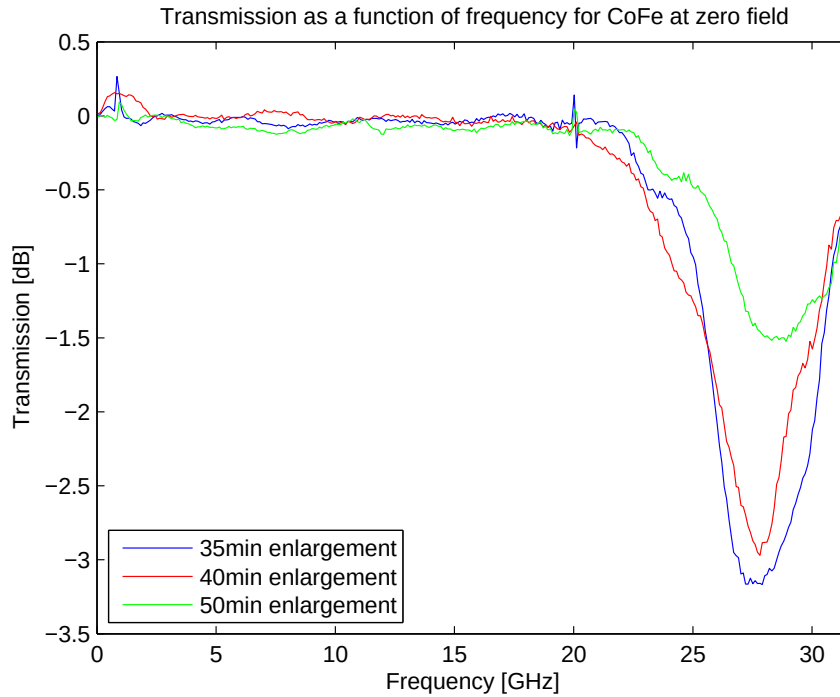


Figure A.6: Transmission vs frequency curve at zero magnetic field for cobalt(55%)-iron(45%) nanowires in an anodized alumina membrane made in UCL with 40min of pore enlargement time.

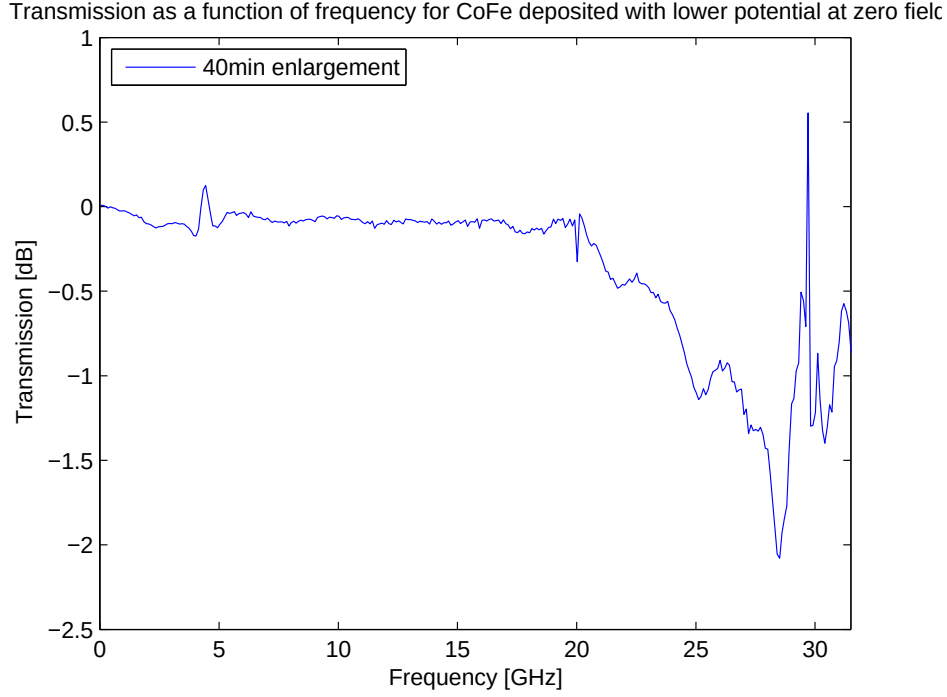


Figure A.7: Transmission vs frequency curve at zero magnetic field for cobalt-iron nanowires deposited with a potential of $-1.5V$ in an anodized alumina membrane made in UCL with $40min$ of pore enlargement time.

First of all, we can see that absorption is indeed observed already for $35min$ of enlargement time. On the graph of CoFe, we clearly see that the peak becomes smaller when the enlargement time is increased. It seems logical since then for the same charge deposited, the nanowires are smaller and intercept less electromagnetic signal. We also see that the peak for CoFe deposited at lower potential ($-1.5V$) is situated at the same frequency than for CoFe deposited at $-0.9V$. The peak is however less precise which would lead to think that the quality of the material is lower.

The most disturbing part is the very large difference between the theoretical and experimental resonance frequencies, the experimental ones being much larger. This can be associated to the Al cathode used for deposition. We have shown that the deposition was slow on aluminum. Hence, if a few nanowires begin to grow a bit faster than others, once there is a ferromagnetic material layer on the aluminum, it becomes a preferential site for nanowires growth. This could lead to the growth of only a few nanowires which would have similar impact on the resonance frequency than a lower porosity. We weren't able to verify this hypothesis at the SEM because the nanowires were not visible in the membrane due to the cutting on the edges and the laser impact on top but it would explain the large shift in frequency. It could also explain why the change in enlargement time doesn't influence the resonance frequency. Indeed, larger enlargement time should induce larger porosity. However, if only a fraction of the nanowires grow, the influence of the porosity on the resonance frequency is much weaker.

Due to these results and the difficulty to remove the aluminum cathode without breaking the alumina, the use of membranes fabricated in UCL was abandoned.

Appendix B

Optical microscope pictures of the membranes containing NiCo nanowires

B.1 Sy

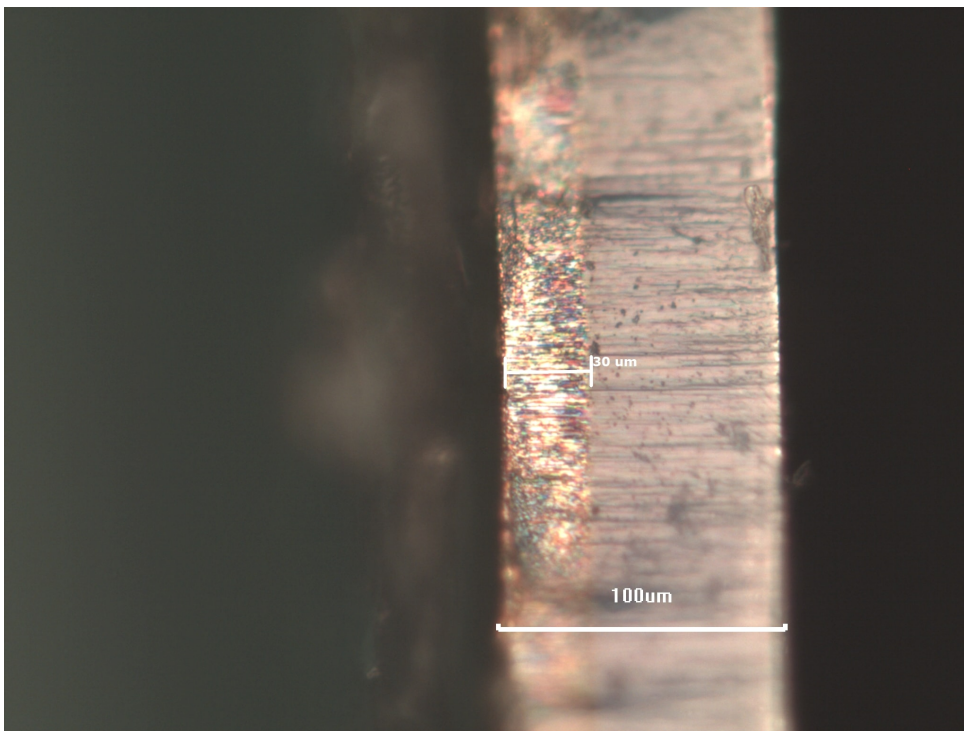


Figure B.1: Optical microscopy sectional view of the Sy filled with high nickel content nanowires $30\mu m$ high on a zone of $10mm$ of diameter (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

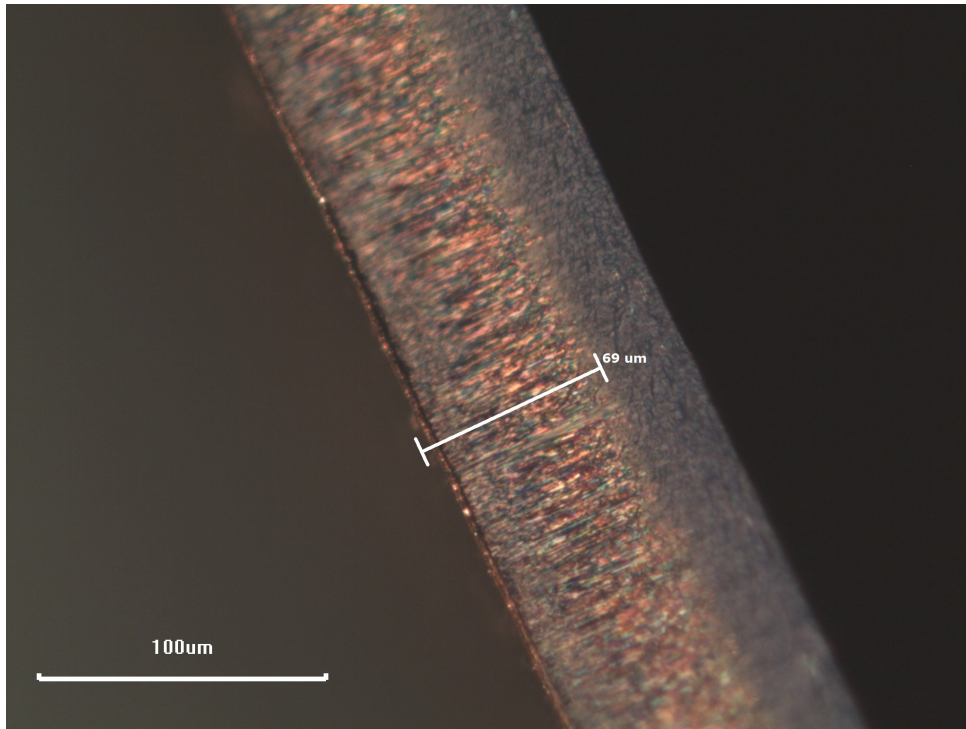


Figure B.2: Optical microscopy sectional view of the Sy filled with high cobalt content nanowires $69\mu\text{m}$ high on a zone of 10mm of diameter (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

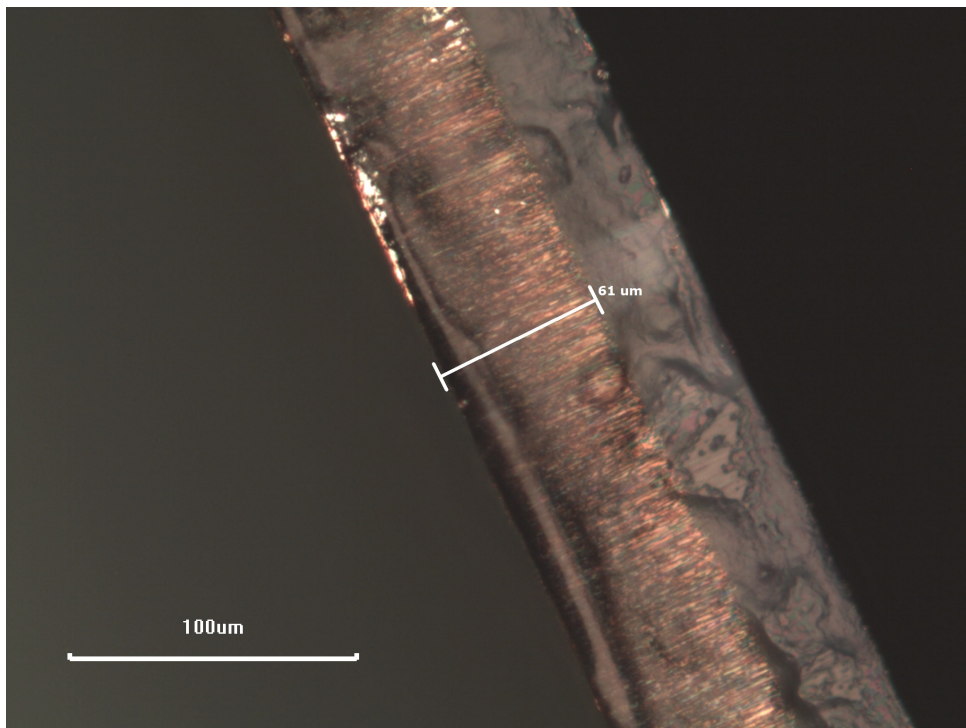


Figure B.3: Optical microscopy sectional view of the Sy filled with high nickel content nanowires $61\mu\text{m}$ high on a zone of 5.9mm of diameter (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

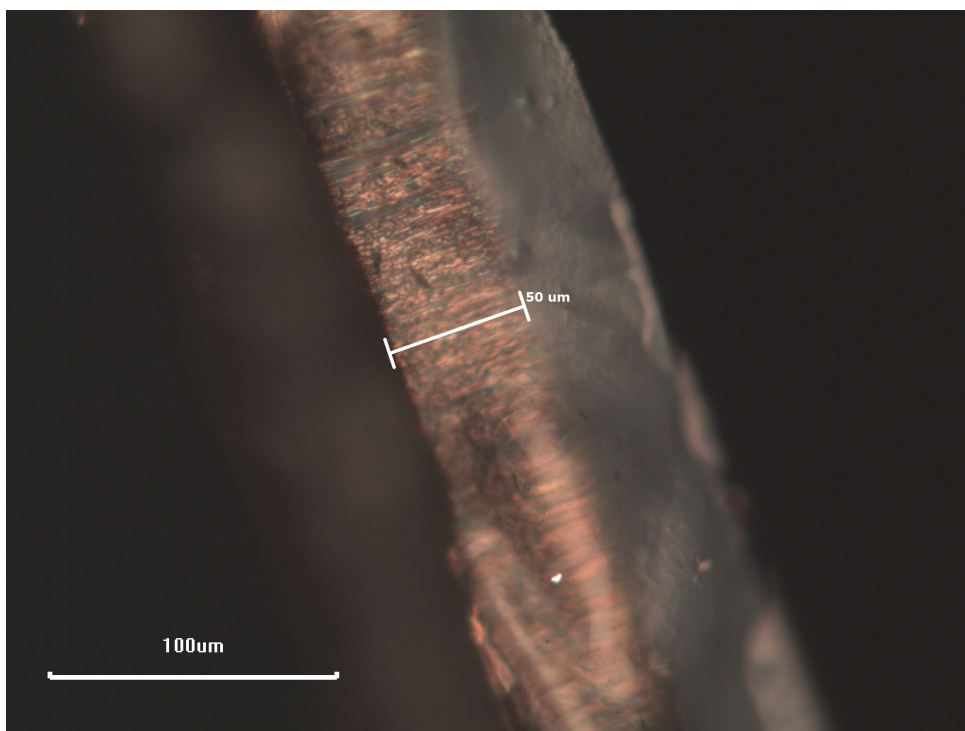


Figure B.4: Optical microscopy sectional view of the Sy filled with high cobalt content nanowires $50\mu\text{m}$ high on a zone of 5.9mm of diameter (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

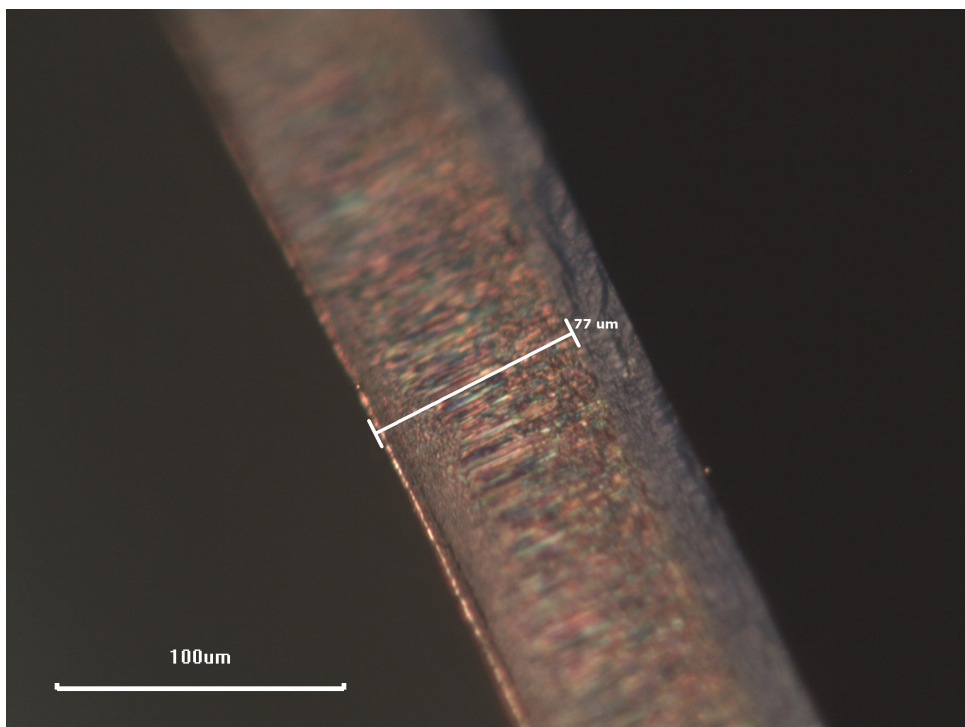


Figure B.5: Optical microscopy sectional view of the Sy filled with high nickel content nanowires $77\mu\text{m}$ high on a zone of 3.9mm of diameter (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

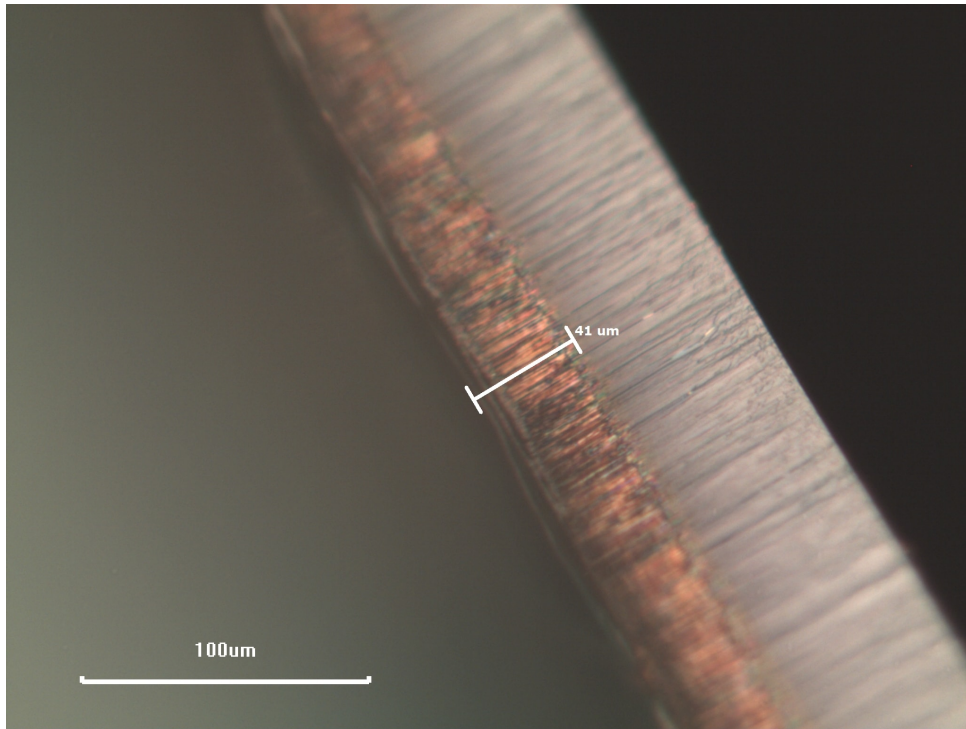


Figure B.6: Optical microscopy sectional view of the Sy filled with high cobalt content nanowires $41\mu m$ high on a zone of $3.9mm$ of diameter (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

B.2 SM

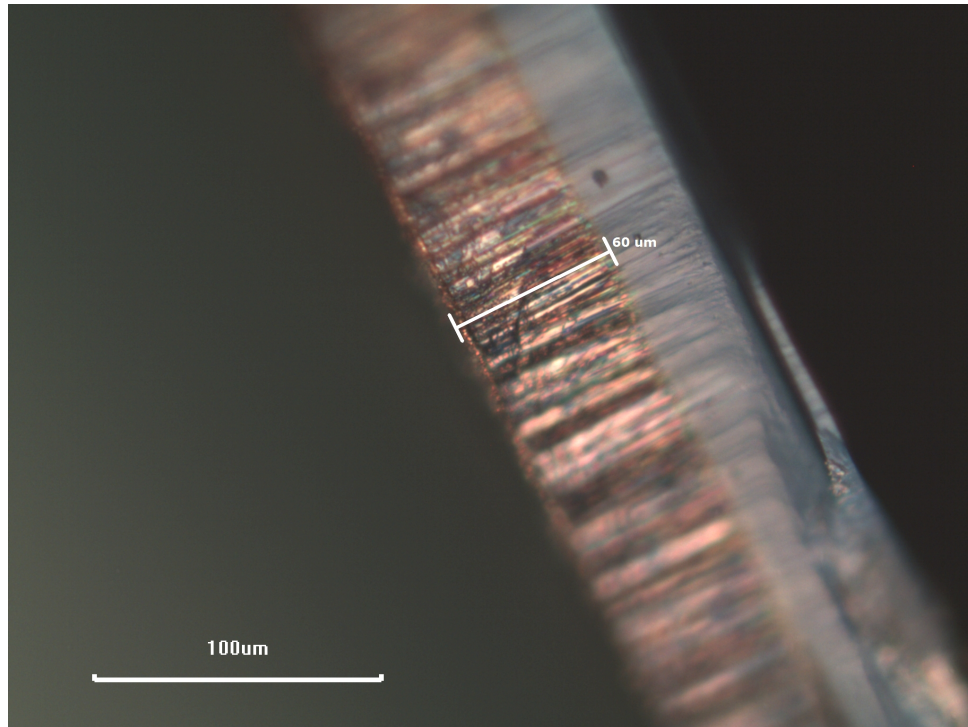


Figure B.7: Optical microscopy sectional view of the Smart membrane filled with high nickel content nanowires $60\mu m$ high on a zone of $11mm$ of length (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

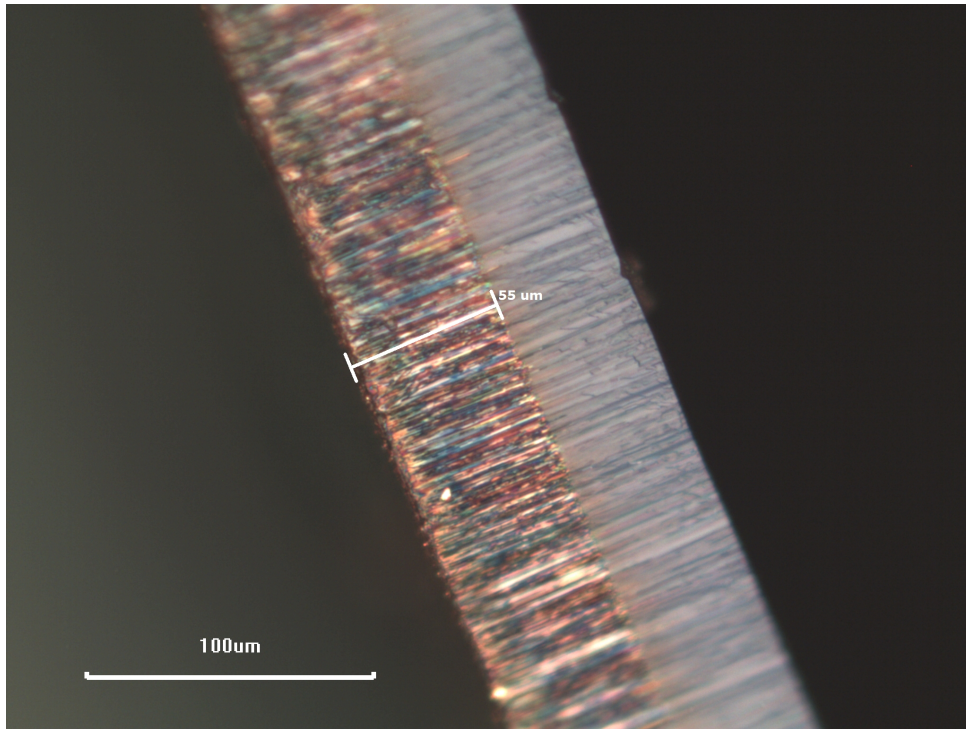


Figure B.8: Optical microscopy sectional view of the Smart membrane filled with high cobalt content nanowires $55\mu m$ high on a zone of $7mm$ of length (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

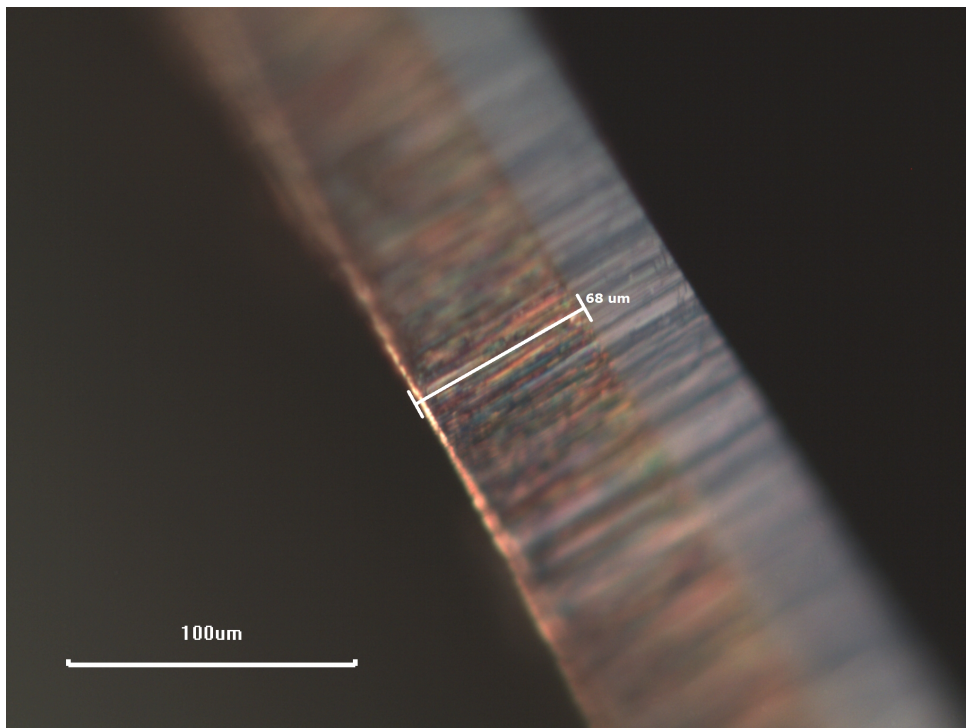


Figure B.9: Optical microscopy sectional view at one end of the Smart membrane filled with high nickel content nanowires $68\mu m$ high on a zone of $25mm$ of length (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

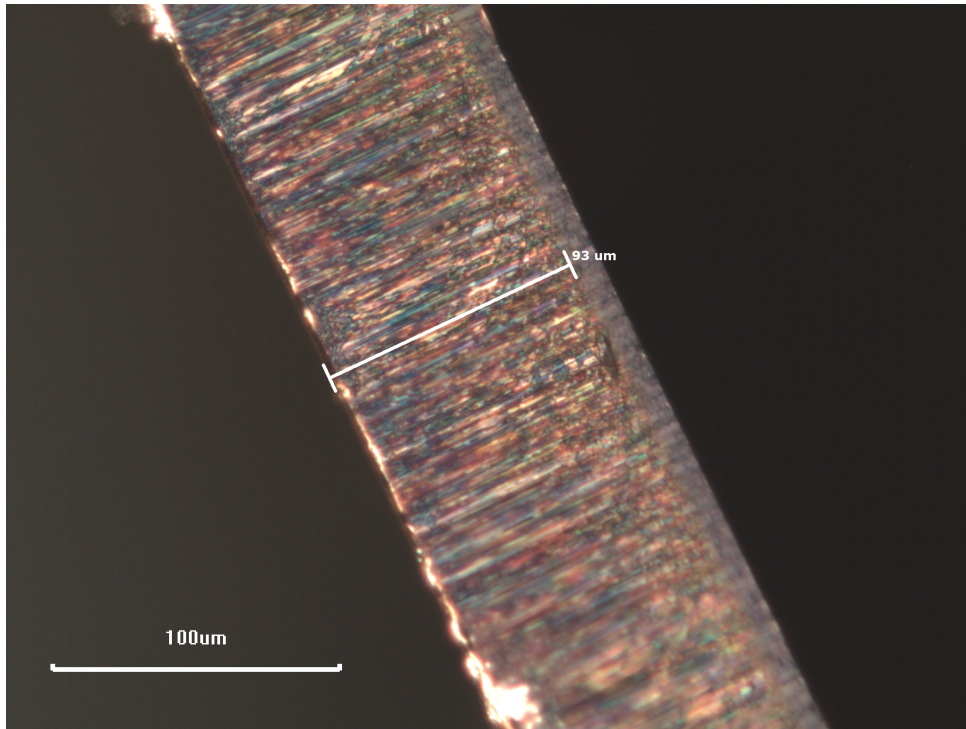


Figure B.10: Optical microscopy sectional view at the middle of the Smart membrane filled with high nickel content nanowires $93\mu\text{m}$ high on a zone of 25mm of length (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

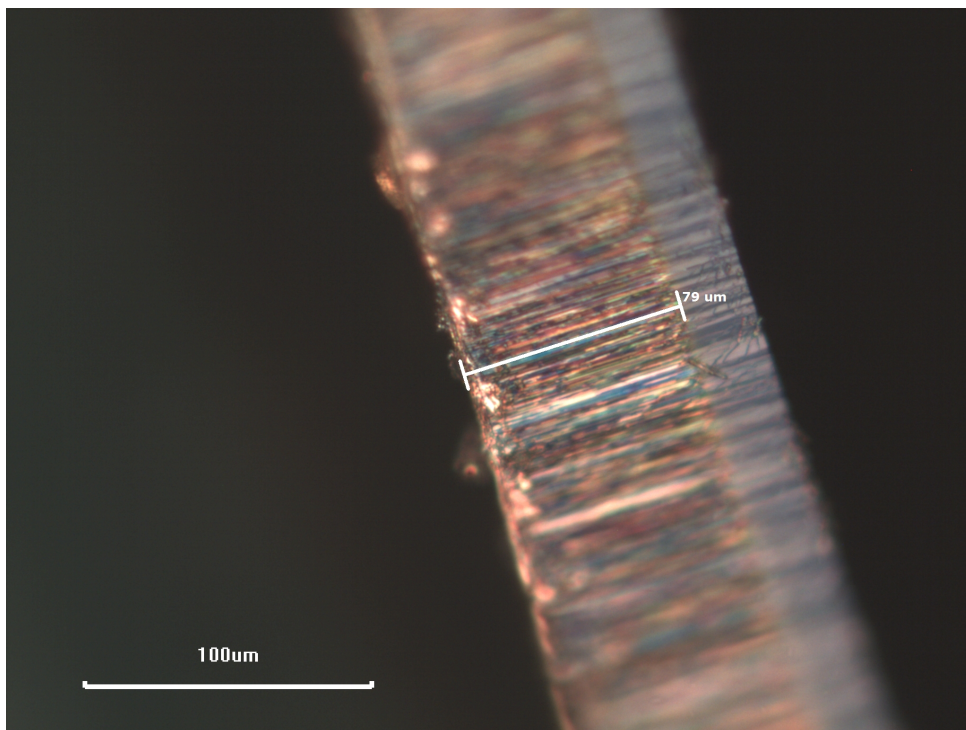


Figure B.11: Optical microscopy sectional view at the other end of the Smart membrane filled with high nickel content nanowires $79\mu\text{m}$ high on a zone of 25mm of length (zoom x20). The nanowires are golden while the empty part of the membrane is grey.

