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Microwave devices based on unbiased tunable ferromagnetic nanowire arrays

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À MJ,
À Timothée,
Et à toutes celles et ceux qui suivront...

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List of Publications

Journal articles

1. Template Approach for Novel Magnetic Ferroelectric Nanocomposites
Luc Piraux, Gaël Hamoir, Ming-Wei Lee, Etienne Ferain, Alain M. Jonas, Isabelle Huynen, and Joaquin De La Torre Medina; Applied Physics Express, Vol. 4, 115001, October 2011
2. Self-Biased Nonreciprocal Microstrip Phase Shifter on Magnetic Nanowired Substrate Suitable for Gyration Applications
Gaël Hamoir, Joaquin De La Torre Medina, Luc Piraux, Isabelle Huynen; IEEE Transactions on Microwave Theory and Techniques, Vol. 60, No. 7, July 2012
3. Q-factor Improvement of Integrated Inductors using High Aspect Ratio Ferromagnetic Nanowires
Gaël Hamoir, Luc Piraux, Isabelle Huynen; Microwave and Optical Technology Letters, Vol. 54, No. 7, July 2012
4. Control of Microwave Circulation using Unbiased Ferromagnetic Nanowires Arrays
Gaël Hamoir, Luc Piraux, Isabelle Huynen; IEEE Transactions on Magnetics, Vol. 49, No. 7, July 2013
5. Influence of the Packing Fraction and Host Matrix on the Magnetoelastic Anisotropy in Ni Nanowire Composite Arrays
Luc Piraux, Gaël Hamoir, Armando Encinas, Joaquin De La Torre Medina, Flavio Abreu Araujo; Journal of Applied Physics, Vol. 114, 123907, September 2013

Conference papers

1. Réseaux de nanofils ferromagnétiques dans l'alumine nanoporeuse: une prometteuse alternative aux ferrites pour les systèmes de communications
Gaël Hamoir, Isabelle Huynen, Luc Piraux; Proceedings of *XIVe Colloque Louis Néel*, p. 2 pages, Brest (France), September 2011
2. Des réseaux de nanofils ferromagnétiques incrustés dans l'alumine nanoporeuse pour les systèmes de communication
Gaël Hamoir, Isabelle Huynen, Luc Piraux; Proceedings of *Journées de Caractérisation Microondes et Matériaux* (JCMM2012), p. 4 pages - Paper ID-16, Chambéry (France), March 2012
3. Unbiased Microwave Circulator using Ferromagnetic Nanowires Arrays
Gaël Hamoir, Isabelle Huynen, Luc Piraux; Proceedings of *12th Joint MMM-Intermag conference*, p. 286, Chicago (USA), January 2013
4. Dispositifs micro-ondes auto-biaisés utilisant des réseaux de nanofils ferromagnétiques
Gaël Hamoir, Isabelle Huynen, Luc Piraux; Proceedings of *XVe Colloque Louis Néel* - p. 92, Tours (France), March 2013
5. Nonreciprocal Microwave Behavior on Magnetic Nanowired Substrates
Joaquin De La Torre Medina, C. E. Carreón González, Gaël Hamoir, Armando Encinas, Luc Piraux, Isabelle Huynen; Proceedings of *International Multidisciplinary Joint Meeting 2013*, p. 1 page, Morelia (Mexico), May 2013

Industrial projects (private reports)

1. Etude du fonctionnement à rémanence de circulateurs planaires à base de réseaux de nanofils ferromagnétiques au sein de membranes d'alumine nanoporeuse
Gaël Hamoir, Luc Piraux, Isabelle Huynen; Rapport d'activité relatif au contrat d'étude entre l'UCL et Thalès Systèmes Aéroportés S.A.; p. 18 pages; June 2012

2. Isolateurs planaires à base de réseaux de nanofils ferromagnétiques au sein de membranes d'alumine nanoporeuse opérant entre 20 GHz et 50 GHz

Gaël Hamoir, Luc Piraux, Isabelle Huynen; Rapport d'activité relatif au contrat d'étude entre l'UCL et Thalès Alénia Space S.A.,

- Volets 1-3; p. 22 pages; February 2013
- Volet 4; p.10 pages; January 2014

Microwave devices are currently used in various areas of telecommunications, from wireless systems to the aerospace industry. Their rapid development over the last few years has brought some issues as the need of enlarging the working range to upper frequencies and the realization of miniaturized and highly integrated components.

The purpose of this thesis is to develop and fabricate microwave devices based on ferromagnetic nanowire arrays embedded in anodic aluminum oxide (AAO) membranes. The magnetic nanowired substrates (MNWS) present several advantages over the ferrites and hexaferrites currently used in microwave devices, in terms of self-biased magnetic materials, design and operation of frequency tunable devices and planar integration. Moreover, the fabrication process, based on electrodeposition, is easy to control and it allows to vary the geometrical and material parameters. The intrinsic properties of MNWS are directly dependent on those geometric and material parameters, such as the length and diameter of the nanowires, the density of the arrays and the choice of the magnetic metal and alloy, which allows a large tunability.

We successfully developed three microwave devices with outstanding properties. Non-reciprocity of the devices is achieved by taking advantage of the anisotropic permeability of the MNWS. First, a nonreciprocal microstrip topology, with specific configurations of nanowire areas in MNWS, is used as an isolator to prevent possible reflections in a *rf* circuit, or as a differential phase shifter used in the phase arrays antennas. Secondly, a nonreciprocal three-ports junction based on the MNWS is developed to fabricate circulators and isolators for airborne or aerospace applications. Finally, microwave planar inductors based on MNWS are a solution for the compactness and the integration requirements of microelectronics industry. Numeric optimizations on geometric parameters of the MNWS are obtained thanks to the good agreement of theoretical models with the experimental results.

The MNWS were characterized using ferromagnetic resonance (FMR) measurements in order to understand their architecture-magnetic property relationship and, therefore, to pave the way for the design of tunable microwave devices. A model is proposed to explain the large shifts in the FMR absorption of Ni nanowire arrays embedded in polymer membrane due to variations in temperature. Moreover a two-stage strain mechanism is proposed for Ni nanowires grown in small diameter AAO template to understand the observed unexpected enhanced magnetic anisotropy. Finally, we present a new route for the fabrication of magnetic-ferroelectric nanocomposites and an electric-field control of the magnetic properties and resonance frequencies of the MNWS.

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General Introduction

Microwave devices are currently used for applications in various areas of telecommunications, from the mobile communications to the spatial-domain. Their rapid development over the last few years has brought some issues, e. g., the need of enlarging the working range upper frequencies and the realization of miniaturized and highly integrated components. For more than half century, the ferromagnetic ceramic materials, also known as ferrites, have been reported as highly important electronic material [1]. Since the development of microwave ferrites in the 1950s [2], current passive microwave devices based on this materials have received great attention and are nowadays employed in a wide spectrum of applications, such as several types of circulators, isolators, magnetostatic resonators, filters, switches, limiters and tunable electrooptic modulators [1, 3, 4, 5, 6, 7, 8]. Several ferrites magnets are used for the microwave applications: bulk and film yttrium garnet (YIG) [9, 10] in single or polycrystalline configurations are widely used in relatively low-power in the L - and X - frequencies band regions. Spinel-type Ni-Zn, Mn-Mg-Zn, and lithium ferrites [11], as well as hexagonal-type barium ferrite [12] are also used in the development of microwave devices.

However, classical devices present limitations since their frequency of operation is usually limited to the X -band due to the low saturation magnetization value of ferrites, and they must be biased by an external magnetic field, which represents a limitation of size reduction. Self-biased hexaferrites may be used for nonreciprocal millimeter-wave devices [8, 13]. They usually require hybrid topology, for example, the hexaferrite disc is mechanically inserted in a circular cavity drilled in a low-loss insulating substrate supporting rf access lines, requiring bonding wires to make the contact between hexaferrite cavity and planar accesses.

The purpose of this thesis is to develop some microwave devices based on an array of ferromagnetic nanowires with outstanding properties as the working frequencies up to 50 GHz, an unbiased magnetic behavior, the possibility to miniaturize the devices and to integrate in microelectronic circuits. This purpose is clearly to replace the classical ferrite

materials in various commercially available microwave applications. In a second hand, this work aims to understand the magnetic properties of specific nanowire arrays to broaden the possibilities of designing microwave devices.

Ferromagnetic nanowire arrays embedded inside a porous anodic aluminum oxide (AAO) membrane have been proved as an excellent alternative route [14] for microwave devices to the classical ferrite-based materials [8]. Arrays of ferromagnetic nanowires offer outstanding properties in the microwave range of frequencies, such as a high operation frequency, a larger saturation magnetization and the ability to work without any external applied magnetic field. Even more, arrays of ferromagnetic nanowires in a regular AAO membrane present full compatibility with micro- and nano-lithography techniques used in integration of devices for system in package or on chip. Thanks to these advantages, telecommunication applications based on nanowired substrate instead of classical ferrites can be conceived.

Fabrication of ferromagnetic nanowire arrays and characterizations of their magnetic and microwave properties are studied for more than 10 years in our group in various domains as the spintronic [15, 16, 17], the superconductivity [18, 19, 20] or the microwaves. Recent works, including the thesis works of M. Darques [21] and J. De La Torre Medina [22] have already investigated the magnetic properties using the ferromagnetic resonance (FMR). These studies have revealed some interesting properties of the ferromagnetic nanowired substrates, such as the dipolar interaction [23, 24, 25], the selection of the structural and magnetic properties of cobalt nanowires by using the appropriate pH of the electrolytic solution [26, 27], the double ferromagnetic resonance in bistable nanowires [28, 29, 30] and the magnetostatic interactions in multi-layered CoCu/Cu nanowires [31, 32]. On the other hand, conception and fabrication of applications using magnetic nanowires inside nanoporous polycarbonate membranes have been investigated in the thesis works of A. Saib [33] and J. Spiegel [34]. Development of theoretical models permit to explain the microwave behavior of the nanowired substrates, including the calculation of the effective permittivity [35].

Their fabrication, easy and not expensive, consists in a bottom-up method using electro-deposition inside nanoporous membranes. This method allows to prepare a variety of nanowire arrays and the nature of magnetic material, as well as both length and packing factor, can be chosen to match the desired magnetic properties. The tunability of their geometric parameters is the main advantage of the nanowired substrate in the development of tunable microwave applications. Other main prop-

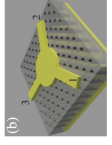
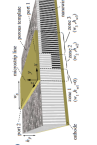
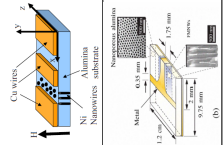
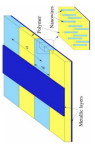
Circulators		Saib <i>et al</i> [36]	PC + Co NWs	$f_w = 26$ GHz Performance=17 dB/cm Insertion loss=11 dB/cm	Self-Biased Hexaferrite [13] $f_w = 28$ GHz Perf.=22 dB/cm Insert. Loss=2.9 dB/cm
		Darques <i>et al</i> [37]	AAO + NiFe Nws	$f_w = [11; 19; 29; 3]$ GHz Perf.= [22; 26.5; 31] dB/cm Insert. loss= [4.4; 6.2; 5.3] dB/cm	
Phase Shifters		De La Torre Medina <i>et al</i> [40]	PC + Ni NWs	$H = 9\text{kOe}$ $f_w = 34$ GHz Perf.=20°/cm Perf.=6 dB/cm Insert. loss=10 dB/cm	CRLH structure [38]; $f_w = 1\text{-}4$ GHz Perf.=110°/cm; Insert. loss>1.2 dB/cm Digital latching ferrite [39]; $f_w = 8$ GHz Perf. =337.5°; Insert. loss=0.5 dB ¹ Polymer/ferrite composite [41]; $f_w = 4.5$ GHz
		Carreon <i>et al</i> [42]	AAO + Ni Nws	$H = 8\text{kOe}$ $f_w = 30$ GHz Perf.=70°/cm Perf.=15 dB/cm Insert. loss=30 dB/cm	Perf. =12°/cm; Insert. loss~3.3 dB/cm Superconductor+ferrite [43]; $f_w = 7$ GHz Perf. =55°; Insert. loss=1.5 dB ¹
Isolator		Kuanr <i>et al</i> [45]	AAO + Ni NWs	$H = 5.6\text{kOe}$ $f_w = 24$ GHz Perf.=6.5 dB/cm Insert. loss=2dB/cm	Ferrite edge-mode [44]; $f_w = 6\text{-}18$ GHz Perf.=2.9 dB/cm; Insert. loss=0.6 dB/cm Ferrite edge-mode [46]; $f_w = 4\text{-}8$ GHz
		Carignan <i>et al</i> [48]	AAO + CoFeB	$f_w = [27.3; 31.2]$ GHz Perf.= [14; 3.6] dB/cm Insert. loss>10 dB	Perf.=5 dB/cm; Insert. loss=0.2 dB/cm Ferrite edge-mode [47]; $f_w = 3\text{-}10$ GHz Perf.>7.5 dB/cm; Insert. loss<0.8 dB/cm Edge-mode+discontinuity [49]; $f_w = 7$ GHz Perf.>12 dB/cm; Insert. loss=0.5 dB/cm
Filters		Saib <i>et al</i> [51]	PC + NiFe NWs	$f_w = [21; 26]$ GHz Perf.= [3; 5] dB/cm	Defect modes [50] $f_w < 10$ GHz; Perf. ~ 12.5 dB/cm Photonic bandgap [52] $f_w < 12.8$ GHz; Perf. ~ 1.9 dB/cm

Table 1 – State of the art of microwave devices based on ferromagnetic nano-wired substrates, with the used membrane and magnetic materials and the most relevant experimental results. The last column presents similar devices based on other structures. ¹Unkonwn length

erty of the nanowired substrates is their nonreciprocity in the microwave range due to the non-diagonal terms of the effective permeability tensor. Such nanowired templates were used over the last years to design various microwave devices [14], such as noise suppressors [53], filters [51], nonreciprocal phase shifters [40, 42], isolators [45, 48], and circulators [36, 54, 37]. The state of the art concerning microwave devices based on ferromagnetic nanowire arrays is listed in table 1 with the membrane and magnetic materials and their most relevant experimental results.

In the first chapter, basic static properties of ferromagnetic nanowire arrays are recorded and the magnetic anisotropies existing in such nanocomposite material are explained. A phenomenological description of the ferromagnetic resonance (FMR) is needed for a correct understanding of the results and the calculation of the effective permeability of a magnetic material. Magnetic anisotropies control the effective field of the nanocomposite and depend on the nature and geometric parameters of nanowire array. Therefore, the microwave properties that depend on the magnetic anisotropy, are tunable with the length or the nature of the nanowires and their packing density [55]. Summarized theoretical background of the microwave devices that are developed in this work, is given at the end of the chapter.

Chapter 2 describes the methods of fabrication of the nanowired substrates and their magnetic, dielectric and geometric characteristics. Theoretical models define the intrinsic properties of an array of nanowires inside any nanoporous templates. The effective permeability is defined as a tensor with non-diagonal terms that are dependent on the FMR frequency, but also on the geometric parameters of the nanowired substrate. The effective permittivity can be calculated taking into account the two-layers system of nanowired substrates and a wide range for the value of dielectric constant allows finding the precise properties for the microwave applications. Relatively-high dielectric loss tangent factor has been observed as the main issue of nanowired AAO substrate on the way to industrial development. Thanks to the model of effective permittivity [35], the high value of loss tangent factor is found to be induced by the high dielectric losses of nanoporous AAO membrane, since the nanowires do not induce any additional losses. Some basic *rf* characterizations on the commercial AAO membrane, including crystallographic structures or consequences of residual moisture in the nanopores, are performed in the purpose to reduce the value of the loss tangent factor and to increase the transmitted signal to the industrial required performances. Other studies on the ohmic losses of transmission lines are accomplished in a

similar goal. Thickness of the lines has to be increased to a certain value to avoid the skin depth effect over all frequency range, and to reduce the roughness value of the lines and enhance the transmitted signal. An additional solution consisting in a composite with nanowired substrate and low-loss dielectric films and showing optimized numerical results is proposed. At the end of the chapter, magnetometer measurements and thermal characterizations of the NiFe and CoFe nanowire arrays are done to observe, respectively, the magnetic remanence which is important for an unbiased nonreciprocal behavior, and the stability of their magnetic and microwave properties at the working spatial temperature range $[-15^{\circ}\text{C} : +65^{\circ}\text{C}]$ or the robustness of the device in the temperature range $[-55^{\circ}\text{C} : +125^{\circ}\text{C}]$ to confirm the ability of these samples to resist to the thermal conditions of spatial applications.

Chapters 3 and 4 present the results of nonreciprocal devices based on self-biased nanowired substrates for two kinds of transmission lines configuration. In Chapter 3, the device is based on a microstrip topology and specific configurations for the nanowire areas in the substrate. Nonreciprocity is achieved by the edge-guided mode induced by the permeability tensor of the substrate, which concentrate the electric and magnetic fields near one long edge of the microstrip. Previous works using the edge-guided mode for nonreciprocal device based on magnetic nanowired substrates already showed differential phase shifts [40, 42]. The proposed device consists of a microstrip-line geometry on a single planar substrate filled with nanowires of different magnetic alloys. Two types of applications can be made with this topology: differential phase shifter can be conceived on the principles that the phase velocity of microwave signal varies in function of the nanowires alloys, or microwave isolation can be achieved at certain frequencies when the signal is absorbed in the forward direction while the signal is transmitted in the reverse direction, due to the edge-guided mode. Experimental confirmation, coupled with good agreements with numerical simulations, demonstrated the nonreciprocal microwave behavior of these samples. For both applications, parametric characterization is numerically performed to optimize the properties or to adjust the working frequencies.

In chapter 4, the device is based on a three-ports junction topology. Nonreciprocity is achieved by the counter-rotating modes in cylindrical cavity [56]. Magnetic nanowires are present below the disk of the three-ports junction and induce the nonreciprocity to the device [36, 54]. At certain frequencies, resonance conditions appear for the clockwise and counterclockwise rotating modes when magnetic substrates are present in the cylindrical cavity. At the circulation frequency, situated between

these two resonant frequencies, the velocity of microwave signal is different for the clockwise and counterclockwise rotating modes. This device can be conceived as a three-port device (circulator) or as a two-ports device (isolator) by applying a short-circuit to the third port. Experimental confirmation and numerical characterization for both device configurations prove the nonreciprocal microwave behavior. Parametric studies are also done to observe the dependence of the working frequency on geometric parameters and to optimize the device.

Both microstrip and three-ports junction configurations are part of projects led in collaboration with *Thalès Systèmes Aéroportés*, and *Thalès Alenia Space* (France) in the purpose to develop microwave devices for airborne or aerospace applications based on the technology built on a ferromagnetic nanowire array.

Chapter 5 summarizes a solution to the compactness and integration requirements of microelectronics industry. The principle is to increase the value of the inductance without increasing the losses. Different solutions are already proposed to increase the quality factor of planar inductors. Either a low-loss substrates, like bulk alumina [57] or porous silicon [58], are used to decrease the losses of the system, or a ferromagnetic core is included in the system to increase the inductance value, with the important drawback that ferromagnetic resonance and eddy currents induced in the volume of the core deteriorate the quality factor at *rf* frequencies. Various technologies were proposed in the literature to overcome those problems. First, a very thin ferromagnetic layer was placed into the core of inductor for increasing its inductance value [59], with the inconvenience that the FMR frequency is usually limited to 1 GHz [59, 60, 61, 62]. Next, modified geometries of the ferromagnetic layer were proposed to increase the shape anisotropy field, hence the FMR frequency, and reduce simultaneously the cross-section accessible to eddy current: slotted [60] or wire/strip patterned [61, 63] ferromagnetic films, micro/nano patterned ferromagnetic cores [62], or nanocomposite cores made of Ni-supported nanoporous alumina membrane [64]. In these topologies, the low amount of ferromagnetic material required to avoid the eddy current together with the attempt to increase the FMR frequency prevents an important increase of permeability [65], hence of the inductance. Reported values are usually lower than 15% and the corresponding quality factor does not follow the inductance enhancement: it is kept unchanged or even lowered. This device is achieved by placing planar inductors on magnetic nanowired substrates that have an enhanced effective permeability by the ferromagnetic resonance below the FMR frequency. Far away the FMR frequency, no additional losses

are induced by the presence of metallic nanowires since their diameter is much smaller than the skin depth [35].

Arrays of ferromagnetic nanowires are also of considerable interest for fundamental physics studies. Understanding of the interactions induced in this type of nanocomposite allows tuning the magnetic and microwave properties of nanowired substrates. The magnetic anisotropies of ferromagnetic nanowire arrays can be controlled by many parameters, such as the nanowire height, diameter or packing fraction of the nanowire array, or as the magnetic material. Models permit to determine the contribution of each anisotropy to the effective resonance field. Magnetostatic anisotropy, including shape anisotropy and dipolar interactions can be expressed as a function of the packing factor [23]. Magnetocrystalline anisotropy in Co nanowire arrays can be described as a function of the pH of electrolyte [27]. Magnetoelastic effects provide an additional parameter to control the magnetic properties by inducing mechanical coupling between the two phases of the nanowired composite. Previous works have demonstrated the large contribution of magnetoelastic anisotropy at low temperature in Ni nanowire arrays embedded in polycarbonate membranes [66, 67, 68] or in porous alumina membrane [69, 70]. However, no consistent overall description of the temperature-dependent magnetic anisotropy in nanowires, that considers both different membrane materials and packing fraction, is available.

In chapter 6, Ni nanowire arrays embedded in various nanoporous templates (polycarbonate, polyvinylidene difluoride and alumina) are characterized for their magnetoelastic effect that arises as well at low temperature than at room temperature. A first study is done at low temperature to observe the dependence of magnetoelastic anisotropy on the membrane material or the packing factor. Theoretical model is proposed to link the large shift of resonance field to the mechanical coupling between nanowires and template. Enhanced anisotropy that was already observed by J. De La Torre Medina [22] in small diameter Ni nanowires embedded in AAO membrane has been investigated and series of measurements, such as diameter characterization, low temperature FMR measurements, X-ray diffraction and alternating magnetometer, have provided information to propose a detailed model. A two-stages system exists in such small diameter nanowired samples, with a compressed part of the wire, which enhances its magnetic anisotropy. A characterization of anisotropy energies allows calculating the strains existing in both stages.

In order to exploit the magnetoelastic effect of Ni nanowires in micro-

wave applications, devices operating at room temperature have to be developed since temperature-controlled environment devices require robust and energy-intensive cryogenic systems. An approach for novel magnetic ferroelectric nanocomposites is proposed in chapter 7. In this new nanocomposite, the magnetoelastic effect arises from the mechanical coupling between a magnetostrictive phase of Ni nanowires and piezoelectric phase of polyvinylidene difluoride template. The large volume to surface ratio that exists in such nanocomposite allows enhancing the multiferroic behavior, which has already been observed in multilayered composite material made of PVDF and ferromagnetic layers [71]. Numerical simulations provide the distribution of dc electric field inside the nanowired substrate and experimental results give the confirmation of a magnetoelectric effect.

Chapter 1

Theoretical background of magnetic properties and microwave devices

1.1 Ferromagnetic material

Magnetic materials naturally exist on earth and have been discovered in the dawn of civilization in China in the form of magnetite [72]. Quickly, the compass has been the first application that humans have conceived with the development of magnetism. Much more after, Maxwell theoretically described in its well-known equations the discovery of Oersted and Ampère, which assumes dependence between electricity and magnetism. These well-known formulas have opened a large range for magnetic applications in the current life.

1.1.1 Basic static properties

The ferromagnetic materials exhibit a large susceptibility, which is the consequence of high and long-range ordering phenomenon (Fig. 1.1). In these regions, the intense magnetic field is induced by the high coupling interactions at the atomic level that align in the same direction all the magnetic moments. These regions are called domains and are randomly oriented which reduces the magnetization of a bulk ferromagnet. In a ferromagnetic material, a small-applied magnetic field is enough to align all the domains and create a magnetization.

A ferromagnet is characterized by some parameters that can be plotted in the hysteresis loop (Fig. 1.2).

After a first magnetization (dashed line), magnetic domains align

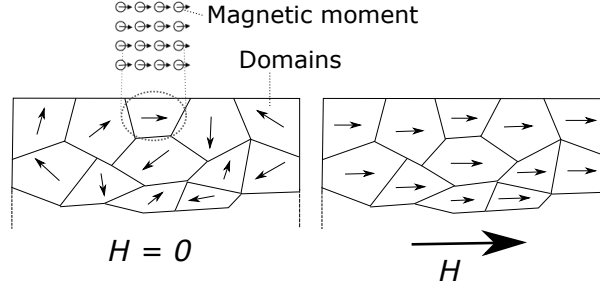


Figure 1.1 – Magnetic domains of a ferromagnet without and with external magnetic field H .

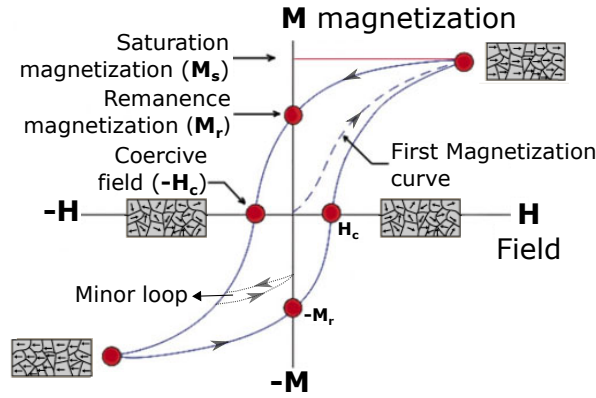


Figure 1.2 – Hysteresis curves.

themselves and the material has reached the saturation magnetization (M_s). When the applied magnetic field decreases until zero, the magnetic field of the material, called remanence magnetization (M_r) is not negligible. Magnetic domains are mainly kept aligned. As driving the applied magnetic field to negative values, the coercive field (H_c) permits to force the magnetization back to zero. Then, if the applied magnetic field is decreasing to large negative values, the material will get a saturation magnetization in the other direction. Finally, the reverse cycle, from negative to positive values, exhibit a same behavior. In a similar way, minor loops (dotted line) can occur if magnetization has not reached its saturation. The hysteresis loop shows the history of the magnetization of the ferromagnetic material.

Table 1.1 gives the experimental values for all ferromagnetic materials that will be used in our experiments, as the g -factor, γ or M_s , respectively the spectroscopic splitting factor, the gyromagnetic ratio and the saturation magnetization. NiFe alloy is only grown with the specific composition $\text{Ni}_{80}\text{Fe}_{20}$, which can be assimilated to the *permalloy* material. CoFe alloy is used with the composition $\text{Co}_{55}\text{Fe}_{45}$. The choice of alloys compositions responds to the need to get material with negligible magnetostrictive and magnetocrystalline properties, contrary to Ni or Fe materials, which show large magnetoelastic or magnetocrystalline anisotropies [73].

	g -factor	γ [GHz·kOe ⁻¹]	M_s [emu·cm ⁻³]
Ni (fcc)	2.20 [74]	3.09	485 [73]
Fe (bcc)	2.088 [75]	2.94	1715 [73]
$\text{Ni}_{80}\text{Fe}_{20}$	2.122	2.97	850 [73]
$\text{Co}_{55}\text{Fe}_{45}$	2.115 [75]	2.96	1917 [73]

Table 1.1 – Values at room temperature (g -factor, gyromagnetic ratio γ and saturation magnetization M_s) for ferromagnetic materials considered in this thesis.

1.1.2 Magnetic anisotropies

The equilibrium direction of magnetization is determined by the minimum energy of the system. Several anisotropy contributions have to be considered in the calculation of the magnetic energy. The origins of these anisotropies are multiple and they can be considered as intrinsic or extrinsic to the magnetic material. The effective magnetic field H_{eff} , linked to the effective energy K_{eff} , is the field related to all the anisotropy and is used in the calculation of ferromagnetic resonance explained in next section. K_{eff} can be defined as the addition of all anisotropy energies:

$$K_{eff} = K_{exch} + K_z + K_{ms} + K_{mc} + K_{me}. \quad (1.1)$$

K_{exch} is the exchange energy, K_z the Zeeman energy, K_{ms} the magnetostatic anisotropy energy, K_{mc} the magnetocrystalline anisotropy energy and K_{me} the magnetoelastic anisotropy energy. Even if K_{exch} , which describes the spin-spin interaction, is at the origin of ferromagnetism, it is not considered in this analysis because of its short-range interaction and its negligible value compared to other energies. Moreover,

we consider the magnetic nanowire arrays in the macrospin approximation. It means that all the spins precess at unison.

The anisotropy contributions that will be used in this thesis are briefly described and short descriptions about the specific geometry of a ferromagnetic nanowire array are given in the boxes.

Zeeman energy

If we apply an external magnetic field $\vec{H}_{ext}$, the magnetization tends to align with the direction of the field. This contribution can be calculated thanks to the following equation [76]:

$$K_z = - \int \vec{H}_{ext} \cdot \vec{M}. \quad (1.2)$$

Magnetostatic anisotropy energy

This anisotropy is dependent on the shape of the sample and, in the case of a sample constituted by multi-separated magnetic elements, it is also dependent on the interaction between all the elements since magnetic interactions are long-range phenomenon. Inside the magnetic material, it exists a demagnetizing field $\vec{H}_{dem}$ which tends to align with the direction of the longest dimension. In the case of a ferromagnet, $\vec{H}_{dem}$ is opposite to the magnetization. We can calculate the magnetic field inside each body by $\vec{H}_i = \vec{H}_{ext} + \vec{H}_{dem}$ where $\vec{H}_{ext}$ is the applied field (Fig. 1.3). $\vec{H}_{dem}$ is a linear function of the magnetization and can be expressed by:

$$\vec{H}_{dem} = -\overline{\overline{N}}\vec{M}, \quad (1.3)$$

where $\overline{\overline{N}}$ is a symmetric 3×3 tensor representing the coefficient of the demagnetizing field. For isotropic material, $\overline{\overline{N}}$ can be written by

$$\overline{\overline{N}} = \begin{bmatrix} N_{xx} & 0 & 0 \\ 0 & N_{yy} & 0 \\ 0 & 0 & N_{zz} \end{bmatrix}, \quad (1.4)$$

with N_{ii} , for $i = x, y, z$, are the demagnetizing factor along the coordinate axis x, y, z respectively and obey the relation

$$N_{xx} + N_{yy} + N_{zz} = 4\pi. \quad (1.5)$$

Depending on the shape of the sample, the magnetostatic anisotropy energy is given by

$$K_{ms} = -\frac{1}{2}\vec{M} \cdot \vec{H}_{dem} = \frac{1}{2}\vec{M} \cdot \overline{\overline{N}}\vec{M}. \quad (1.6)$$

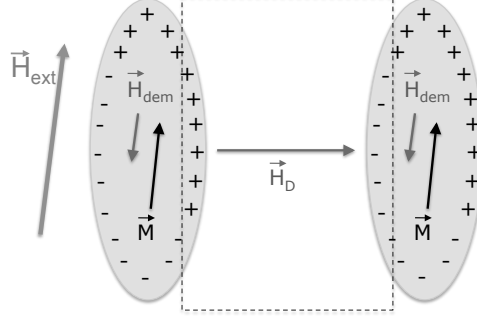


Figure 1.3 – Schematic representation of two identical magnetized bodies interacting each other. A demagnetizing field $\vec{H}_{dem}$ appears inside each body and a dipolar field $\vec{H}_D$ is induced between bodies.

In the case of a sample constituted by multi-separated magnetic elements, dipolar magnetic interactions have to be considered. A dipolar field $\vec{H}_D$ can be observed between two magnetic elements due to the coupling interactions of the *magnetic charges* on the surface of each element. Dipolar interactions energy can be calculated in the particular case of two bodies i and j , with the same volume V but not necessarily the same shape, by the formula:

$$K_{D_{ij}} = -\vec{M}_i \cdot \vec{H}_{D_{ij}}, \quad (1.7)$$

where $\vec{M}_{i,j} = M_s V \vec{m}_{i,j}$ are the magnetizations for the bodies i and j respectively, $\vec{m}_{i,j}$ are the unit vectors and $\vec{H}_{D_{ij}}$ and $\vec{H}_{D_{ji}}$ are the dipolar field experienced by each body due to the presence of the other. $\vec{H}_{D_{ij}}$ can be calculated thanks to the interdistance of the elements $\vec{r}_{ij} = \vec{r}_i - \vec{r}_j$:

$$\vec{H}_{D_{ij}} = \frac{3\vec{r}_{ij} (\vec{r}_{ij} \cdot \vec{M}_j)}{\|\vec{r}_{ij}\|^5} - \frac{\vec{M}_j}{\|\vec{r}_{ij}\|^3}. \quad (1.8)$$

Eq. (1.8) clearly demonstrates the dependence of $H_{D_{ij}}$ on the separation distance between both elements. The total dipolar energy of N elements is:

$$K_D = \frac{1}{2} \sum_{i=1}^N \sum_{j=1}^N K_{D_{ij}}. \quad (1.9)$$

In the case of ferromagnetic nanowire arrays, nanowires can be considered as infinite cylinders due to the large aspect ratio. Due to the large number of nanowires in an array, the mean field approach is usually used instead of eq. (1.9), which requires time-consuming calculations. The mean field approach is expressed as a linear combination between an isolated infinite cylinder and a homogeneous infinite film, for which the demagnetizing tensors are known. The magnetostatic anisotropy energy is then the addition of terms of both particular cases, with the dependence of the packing factor P of the nanowire array.

$$K_{ms} = \frac{1}{2} (1 - P) \vec{M} \cdot \overline{\overline{N}}_w \vec{M} + \frac{P}{2} \vec{M} \cdot \overline{\overline{N}}_f \vec{M}, \quad (1.10)$$

where $\overline{\overline{N}}_w$ and $\overline{\overline{N}}_f$ are the demagnetizing factor for an isolated infinitely long wire and an infinite film, and $\vec{M} = M_s \vec{m}$, the saturation magnetization and $\vec{m}$ the magnetization unit vector. Packing fraction of the nanowire array, associated to the porosity of the membrane, is calculated as the product of the pores density ρ and the section area of a nanowire $A_p = \pi d^2/4$, with d the nanowire diameter. Eq. 1.10 display the both particular cases, when $P = 0$ or $P = 1$, the magnetostatic energy is given either by $\overline{\overline{N}}_w$ or $\overline{\overline{N}}_f$.

The demagnetizing factor of a infinite cylinder and for infinite film, for a axis wire parallel to the z -axis or perpendicular to the film, is equivalent to:

$$\overline{\overline{N}}_w = \begin{bmatrix} 2\pi & 0 & 0 \\ 0 & 2\pi & 0 \\ 0 & 0 & 0 \end{bmatrix}, \quad \overline{\overline{N}}_f = \begin{bmatrix} 0 & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 4\pi \end{bmatrix}. \quad (1.11)$$

Substituting the demagnetizing factors to eq. (1.10), the magnetostatic energy becomes:

$$K_{ms} = 2\pi P M_s^2 + \pi M_s^2 (1 - 3P) \sin^2 \theta, \quad (1.12)$$

where θ is the angle of $\vec{M}$. More details on the calculation of magnetostatic energy can be found in the thesis of Dr. J. De La Torre Medina [22].

In analogous manner, the magnetostatic field H_{ms} of a nanowire array can be found as a linear combination of the self demagnetizing field for both isolated infinite cylinder and the infinite film. H_{ms} is also dependent of the packing factor P :

$$H_{ms} = 2\pi M_s (1 - 3P). \quad (1.13)$$

The magnetocrystalline anisotropy

In several crystallographic structures, the electronic orbitals are deformed by their interactions with other electrons and tend to align in the easy direction. The main cause of magnetocrystalline anisotropy is the spin-orbit coupling, but some other interactions have to be taken into account to determine the magnetic energy from this contribution [76]. The spins motion induces a change in the shape of atomic electrons, which stretch into one direction. Another contribution to the magnetocrystalline anisotropy could be the magnetic (dipole-dipole) coupling of elementary moments that appears when the magnetic moments form a lattice and have the same values and orientations. This contribution is dependent on the orientation of the moments to the lattice axes. A third origin of magnetocrystalline anisotropy should be considered in the terms of one-ion origin. The ions have their own energy spectrum, characterized by the energy levels. The free-energy density, which can be linked to the magnetic energy, is calculated thanks to the Boltzmann distribution representing the sum of all ions energy levels. Those models show good agreement with experimental results but should not be considered as independent mechanisms.

Thanks to the phenomenological description, the expressions of energy of the magnetocrystalline anisotropy can be written down in the form of power series. Anisotropy constants are used for the coefficients of the series and this expression is dependent to the symmetry of crystal lattice. Only odd power terms is kept on the expression to preserve the invariance of energy. This expression takes the form:

$$K_{mc} = K_1^{(1)} \alpha_1^2 + K_1^{(2)} \alpha_2^2 + K'_{mc}, \quad (1.14)$$

with α_j the direction cosines of $\vec{M}$ with respect to the symmetry axes, $K_1^{(j)}$ the magnetocrystalline constant and K'_{mc} the sums of other higher power terms. The condition $\alpha_1^2 + \alpha_2^2 + \alpha_3^2 = 1$ gives the omission of one term $K_1^{(j)} \alpha_j^2$. In first approximation, we can neglect the anisotropy in the basal plane (perpendicular to the main axis), eq. (1.14) becomes:

$$E_{mc} = K_1 \sin^2 \theta + K_2 \sin^4 \theta + K_3 \sin^6 \theta + \dots, \quad (1.15)$$

where θ is the angle between $\vec{M}$ and the main axis.

Nickel and iron have both cubic crystallographic structures. As mentioned the Fig. 1.4, the easy-direction of the magnetization for Fe (bcc structure) is (100) and for Ni (fcc structure) (111).

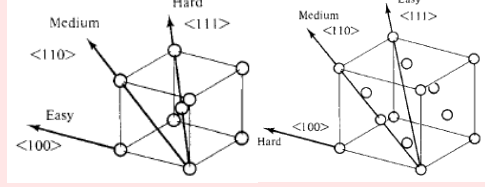


Figure 1.4 – Crystallographic structures of (a) iron (bcc structure) and (b) nickel (fcc structure) [73].

For cubic crystal, the expression (1.15) can be written as:

$$E_{mc} = K_1 (\alpha_1^2 \alpha_2^2 + \alpha_2^2 \alpha_3^2 + \alpha_3^2 \alpha_1^2) + K_2 \alpha_1^2 \alpha_2^2 \alpha_3^2 + \dots, \quad (1.16)$$

where $\alpha_{1,2,3}$ are the direction cosines of $\vec{M}$ with respect to the directions [100], [010] and [001]. By replacing the angles θ and φ into the directions cosines, we obtain the equation in the spherical coordinates:

$$E_{mc} = \frac{1}{4} K_1 (\sin^2 2\theta + \sin^4 \theta \sin^2 \varphi) + \frac{1}{16} K_2 \sin^2 \theta \sin^2 2\theta \sin^2 2\varphi. \quad (1.17)$$

Fig. 1.5 shows the dependence in temperature of K_1 and K_2 magnetocrystalline energy constants for Ni material [77]. At room temperature, the values of K_1 and K_2 are respectively $-5.2 \cdot 10^4$ erg·cm⁻³ and $-2.2 \cdot 10^4$ erg·cm⁻³. At liquid nitrogen temperature, their absolute values increased by an order of magnitude.

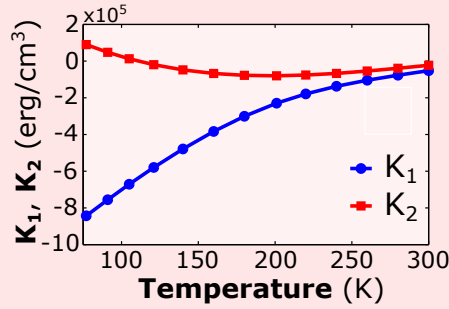


Figure 1.5 – K_1 (blue line) and K_2 (red line) magnetocrystalline constants for Ni in a temperature spectra from liquid nitrogen (77 K) to room temperature (300 K).

The magnetoelastic anisotropy

The magnetoelastic contribution appears in a magnetostrictive material under strain [73]. The magnetostriction is the property of some materials to change their shape according to their magnetization. The opposite phenomenon, called Villary effect, lies in the change of magnetization of materials deformed by an external force. When the magnetization is saturated, the magnetostriction consists in a modification of the length in the direction defined by the directional cosines β_i , for $i = x, y, z$, when the material is magnetized in a direction defined by the directional cosines α_i , for $i = x, y, z$.

The magnetoelastic energy can be calculated taking into account the magnetostriction coefficient λ and the stress σ induced on the magnetic material:

$$K_{me} = |\sigma|\lambda, \quad (1.18)$$

where σ is negative in the case of compressive stress or positive for tensile stress and can be calculated by using the Young modulus E and the nonelastic strain $\Delta\epsilon = \Delta\alpha\Delta T$ resulting of the mismatch between the two elongations of substrate and magnetostrictive material. $\Delta\alpha$ and ΔT are the mismatch in the thermal expansion coefficients and the delta of temperature, respectively.

$$\sigma = \Delta\epsilon E. \quad (1.19)$$

The magnetostriction coefficients for cubic ferromagnets, as Ni or Fe, are described in first approximation by:

$$\begin{aligned} \lambda = & \frac{3}{2}\lambda_{100} \left(\alpha_x^2\beta_x^2 + \alpha_y^2\beta_y^2 + \alpha_z^2\beta_z^2 - \frac{1}{3} \right) \\ & + 3\lambda_{111} (\alpha_x\alpha_y\beta_x\beta_y + \alpha_y\alpha_z\beta_y\beta_z + \alpha_z\alpha_x\beta_z\beta_x), \end{aligned} \quad (1.20)$$

where α_i and β_i , for $i = x, y, z$, are the directional cosines for $\vec{m}$ and $\vec{\sigma}$, respectively. The quantities λ_{100} and λ_{111} are the magnetostrictive fractional elongations in the direction of the [100] and [111] crystallographic axes, respectively. The Ni magnetostriction coefficients λ_{100} and λ_{111} are negative (Fig. 1.6) and have a weak dependence on the temperature [78].

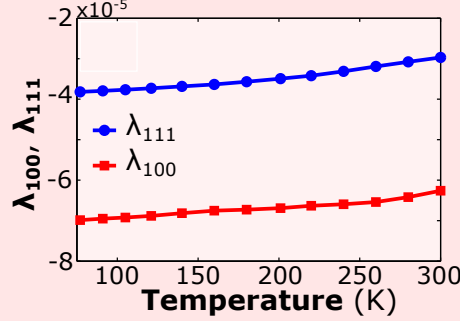


Figure 1.6 – Magnetostriction coefficients λ_{100} (red line) and λ_{111} (blue line) for Ni as a function of temperature.

1.2 Phenomenological description of ferromagnetic resonance

The ferromagnetic resonance is a powerful technique of characterization used to probe the magnetic properties of the materials. It has been discovered by Griffiths in 1946 [79] in a study of the variation in the quality factor of a microwave guide with one side covered by iron, cobalt and nickel thin films : the value of the resonance frequency was too high to explain it with the Larmor equation. Kittel has given the first theoretical explanation in 1947 [80], which gave an interpretation of the anomalous Larmor frequencies observed in Griffiths' experiments. Then he extended his theory to include the effect of the shape and the crystal orientation of the sample [81]. Smit and Beljers [82], and Suhl [83] in 1955 derived the general classical FMR equation which, when is coupled with the energy contributions K of the system, are used to determine specific resonance conditions. The formula of Smit-Suhl allows to calculate the angular frequency resonance ω_0 :

$$\omega_0 = \frac{\gamma}{M_s \sin \theta_0} (K_{\theta\theta} K_{\varphi\varphi} - K_{\theta\varphi}^2)^{1/2}, \quad (1.21)$$

where

$$K_{\theta\theta} = \frac{\partial^2 K}{\partial \theta^2}, \quad K_{\varphi\varphi} = \frac{\partial^2 K}{\partial \varphi^2}, \quad K_{\theta\varphi} = \frac{\partial^2 K}{\partial \theta \partial \varphi}. \quad (1.22)$$

θ and φ are angles determining the direction of the magnetization (Fig. 1.7).

It is worth mentioning that the ferromagnetic resonance in a macroscopic conducting magnetic sample is hidden by huge eddy-current effects that conductively absorb the signal. Small objects have to be made

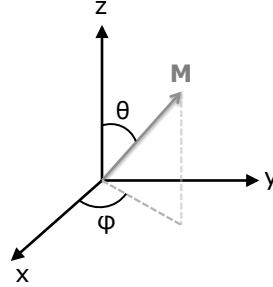


Figure 1.7 – Definition of coordinate system and angles of magnetization.

in the nanometric range to avoid this undesirable effect. The ferromagnetic resonance allows to characterize various magnetic properties of the sample, like the magnetization saturation, the magnetic anisotropy contributions, the gyromagnetic ratio or the relaxation mechanisms of the magnetization [76, 84]. In addition to understand the fundamental problem of interactions of the electromagnetic field with the matter, this technique is of interest as a basis for designing tunable microwave devices that are used now or will be used in the future in telecommunications.

Considering the phenomenological description, the spins of all magnetic moments are precessing around an equilibrium direction at the frequency f_L called Larmor frequency, depending on the gyromagnetic factor γ of the material. A local magnetic field that results on the magnetization contributions of the sample or on the external applied magnetic field will maintain the magnetization of the spins at the equilibrium. If we add oscillating magnetic field from a microwave signal, this *ac* magnetic field will perturb the magnetization of each spin that will diverge of its equilibrium direction. The damping factor will lower the precession of the magnetization that will go back to its equilibrium point. Applying a microwave signal with the same frequency than the precession of the material will maintain the precession of the magnetization, then the resonance condition is fulfilled and the microwave signal is absorbed by the magnetic material (Fig. 1.8).

1.2.1 Equation of motion

Landau-Lifshitz described in 1935 [85] the equation of motion for the magnetization. We first consider the simple form of the equation in the case of uniform oscillations of magnetization in an isotropic ferromagnet

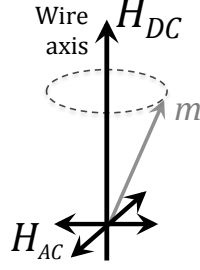


Figure 1.8 – Sketch of ferromagnetic resonance where the magnetization m of the wire is precessing around its equilibrium axis formed by the external magnetic field H_{dc} by the action of ac magnetic field H_{ac} .

without losses for the dissipation of magnetic energy. Thus, the proposed equation is valid strictly for uniform magnetization and in the approximation of a sufficiently slowly motion in space of $\vec{M}$. This equation can be described in a classical approximation even if the magnetization dynamics between the magnetization $\vec{M}$ and the external magnetic field $\vec{H}$ are quantum phenomena.

$$\frac{\partial \vec{M}}{\partial t} = -\gamma \vec{M} \times \vec{H}. \quad (1.23)$$

$\gamma = -g\mu_B/h$ is the gyromagnetic ratio that represents the collective motion of magnetic moments. Its value is related to the g -factor which is very near to the spin value of 2.

An important condition for the equation (1.23) is the conservation of the vector $\vec{M}$ magnitude. The scalar multiplication of (1.23) by $\vec{M}$ gives the condition :

$$\frac{\partial \vec{M}^2}{\partial t} = 0. \quad (1.24)$$

In real ferromagnets, the oscillations of magnetization are mainly coupled with a dissipation of their magnetic energy that transforms into other kinds of energy. We should adapt the equation 1.23 to include a term explaining the dissipation :

$$\frac{\partial \vec{M}}{\partial t} = -\gamma \vec{M} \times \vec{H} - \alpha \frac{\gamma}{M} \vec{M} \times (\vec{M} \times \vec{H}). \quad (1.25)$$

The second term in the right part of the equation represents the magnetic losses in the motion of magnetization. The dimensionless parameter α is the damping factor and in the case of a linear approximation (small amplitudes).

1.2.2 High-frequency magnetic susceptibility

The magnetic susceptibility components of a ferromagnet can be calculated thanks to the equations of motion (1.24) and (1.25) using the approximation of small amplitude of precession. We assume that the magnetization and the magnetic field are the sums of steady and alternating parts:

$$\vec{M} = \vec{M}_0 + \vec{m}_\sim \quad \vec{H} = \vec{H}_0 + \vec{h}_\sim. \quad (1.26)$$

The approximation of small amplitude leads to get alternating parts much smaller than the steady parts.

$$h_\sim \ll H_0 \quad m_\sim \ll M_0. \quad (1.27)$$

First, we resolve the equation of motion without dissipation of magnetic energy. We replace the equations (1.26) into (1.23), considering the zero approximation, the directions of each steady component of $\vec{M}$ and $\vec{H}$ coincide each other and are determined by:

$$\vec{M}_0 \times \vec{H}_0 = 0. \quad (1.28)$$

In the first approximation (called linearization), we neglect the products of alternating quantities $\vec{m}_\sim$ and $\vec{h}_\sim$. Taking into account (1.28), the equation (1.23) becomes:

$$\frac{\partial \vec{m}_\sim}{\partial t} + \gamma \vec{m}_\sim \times \vec{H}_0 = -\gamma \vec{M}_0 \times \vec{h}_\sim. \quad (1.29)$$

To solve the equation (1.29), we should assume that the time dependence of $\vec{h}_\sim$ is sinusoidal, and by the linearity of the equation, time dependence of $\vec{m}_\sim$ will also be sinusoidal. We define $\vec{m}_\sim$ and $\vec{h}_\sim$ as complex number

$$\dot{\vec{m}} = \vec{m} \exp(i\omega t) \quad \dot{\vec{h}} = \vec{h} \exp(i\omega t) \quad (1.30)$$

Replacing (1.30) in the equation (1.29) will give:

$$i\omega \vec{m} + \gamma \vec{m} \times \vec{H}_0 = -\gamma \vec{M}_0 \times \vec{h}. \quad (1.31)$$

Assuming that the direction $\vec{H}_0$ and $\vec{M}_0$ coincides with the z -axis, the equation (1.31) can be expressed in the three equations:

$$\begin{aligned} i\omega m_x + \gamma H_0 m_y &= \gamma M_0 h_y, \\ -\gamma H_0 m_x + i\omega m_y &= -\gamma M_0 h_x, \\ i\omega m_z &= 0. \end{aligned} \quad (1.32)$$

The solution of the system (1.33) is:

$$\begin{aligned} m_x &= \chi h_x + i\chi_a h_y, \\ m_y &= -i\chi_a h_x + \chi h_y, \\ m_z &= 0. \end{aligned} \quad (1.33)$$

χ and χ_a are the diagonal and non-diagonal components of the susceptibility tensor that expressed the following relation $\vec{m} = \bar{\bar{\chi}} \cdot \vec{h}$:

$$\bar{\bar{\chi}} = \begin{bmatrix} \chi & i\chi_a & 0 \\ -i\chi_a & \chi & 0 \\ 0 & 0 & 0 \end{bmatrix}, \quad (1.34)$$

and are equivalent to:

$$\chi = \frac{\gamma M_0 \omega_H}{\omega_H^2 - \omega^2}, \quad \chi_a = \frac{\gamma M_0 \omega}{\omega_H^2 - \omega^2}, \quad (1.35)$$

with $\omega_H = \gamma H_0$.

Thanks to the equations (1.33), we are able to know how $\vec{h}$ interacts with the magnetization $\vec{m}$ of an isotropic and lossless ferromagnet. Each component of $\vec{m}$ perpendicular to its directions of magnetization saturation, is excited by the *ac* $\vec{h}$ perpendicular field but also by the *ac* $\vec{h}$ parallel field. This feature of the equation of motion gives a non-symmetric magnetic susceptibility tensor, which explains the behavior of isotropic magnets.

The susceptibility tensor has a point of resonance when $\omega = \omega_H$, the value of χ and χ_a diverge. It means that $\bar{\bar{\chi}}$ has a dependence on the frequency ω , on the magnetic material (γ , M_s) and on the steady magnetic field amplitude H_0 .

Considering now a ferromagnet with magnetic losses, the approximations (1.26) and (1.27) can be applied in the equation (1.25). In the case of small amplitudes, $\alpha^2 \ll 1$ and the linearization of the equation gives the equation:

$$i\omega \vec{m} + \gamma \vec{m} \times \vec{H}_0 + \frac{i\alpha\omega}{M_0} \vec{m} \times \vec{M}_0 = -\gamma \vec{M}_0 \times \vec{h}. \quad (1.36)$$

The susceptibility tensor components will change to take into account the damping effect of magnetization : $\omega_H \rightarrow \omega_H + i\alpha\omega$, $\chi \rightarrow \chi' + i\chi''$, and $\chi_a \rightarrow \chi'_a + i\chi''_a$. The solution of the linear equation (1.36) allows to

determine the susceptibility components:

$$\begin{aligned}
 \chi' &= \frac{1}{D} \gamma M_0 \omega_H [\omega_H^2 - (1 - \alpha^2) \omega^2], \\
 \chi'' &= \frac{1}{D} \alpha \gamma M_0 \omega [\omega_H^2 + (1 + \alpha^2) \omega^2], \\
 \chi'_a &= \frac{1}{D} \gamma M_0 \omega [\omega_H^2 - (1 + \alpha^2) \omega^2], \\
 \chi''_a &= 2\alpha \gamma M_0 \omega^2 \omega_H, \\
 D &= [\omega_H^2 - (1 + \alpha^2) \omega^2]^2 + 4\alpha^2 \omega^2 \omega_H^2.
 \end{aligned} \tag{1.37}$$

The damping of the precession will change the condition of resonance:

$$\omega = \frac{\omega_H}{1 + \alpha^2}. \tag{1.38}$$

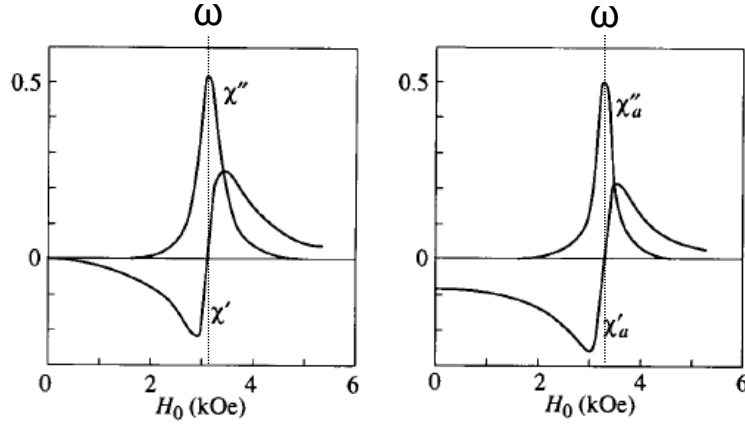


Figure 1.9 – Real and imaginary parts of the tensor $\bar{\bar{\chi}}$ as a function of H_0 calculated using (1.37) with $M_0 = 160$ G, $f = 9.4$ GHz and $\alpha = 0.025$ [76].

At resonance, the real part of the susceptibility will change of sign while the imaginary part will pass through a maximum (Fig. 1.9). The smaller the damping factor is, the higher will be the absorption of the signal. Other change in the susceptibility tensor is the apparition of the z -component (parallel to the magnetization):

$$\bar{\bar{\chi}} = \begin{bmatrix} \chi & i\chi_a & 0 \\ -i\chi_a & \chi & 0 \\ 0 & 0 & \chi_{||} \end{bmatrix}. \tag{1.39}$$

$\chi_{\parallel}$ remains small in the case of small losses and is negligible.

The linewidth ΔH or $\Delta\omega$ of the curve of the absorption can also be calculated by the previous equation, knowing that a high damping factor results in a wide linewidth. ΔH and $\Delta\omega$ are deduced from the condition $\chi'' = \chi''_{\text{res}}/2$.

$$\Delta H = \frac{2\alpha\omega}{\gamma} \quad \Delta\omega = 2\alpha\omega. \quad (1.40)$$

1.2.3 High-frequency permeability

We know that the permeability can be calculated thanks to the relation $\vec{b} = \bar{\vec{\mu}}\vec{h}$, with $\vec{b}$ representing the complex amplitude of *ac* magnetic induction.

$$\bar{\vec{\mu}} = \bar{\vec{I}} + 4\pi\bar{\vec{\chi}} = \begin{bmatrix} \mu & i\mu_a & 0 \\ -i\mu_a & \mu & 0 \\ 0 & 0 & 1 \end{bmatrix}, \quad (1.41)$$

where $\mu = 1 + 4\pi\chi$ and $\mu_a = 4\pi\chi_a$ are the diagonal and non-diagonal parts of the tensor. In the high frequency range, a non-reciprocal behavior appears due to the μ_a term of the permeability tensor, which ensures an inhomogeneous distribution of the *ac* fields in the magnetic sample.

1.3 Principles of microwave devices

In this section, we described several microwave devices that will be developed in this thesis. Explanation of the principle and the purpose for each device is given. For nonreciprocal devices, the principle can be defined by the matrix of *S*-parameters which gives the reflection and transmission coefficients of the signal propagation. It writes as

$$S = \begin{bmatrix} S_{11} & S_{12} & \dots \\ S_{21} & \dots & \dots \\ \dots & \dots & S_{ii} \end{bmatrix}, \quad (1.42)$$

with *i* the number of ports in the device.

1.3.1 Isolator

A microwave isolator is a nonreciprocal two-ports device that transmits the microwave signal only in one direction of propagation. The *S*-parameters are represented by a 2×2 -matrix

$$S = \begin{bmatrix} 0 & 0 \\ 1 & 0 \end{bmatrix}, \quad (1.43)$$

with only the S_{21} parameters not equal to zero. It means that the signal power flows only from the port 1 to the port 2.

Several configurations (Fig. 1.10) of isolator can be conceived as the resonance isolator or the field displacement isolator [86]. The field displacement isolators (Fig. 1.10(a)) use ferrite slab situated vertically in a specific place inside the waveguide. Microwave electric fields from forward and reverse propagation direction are quite different, which causes the attenuation of the signal in the reverse direction. Resonance isolators (Fig. 1.10(b)) use ferrite slab situated in the optimum location of the waveguide where the rf signal will be absorbed by the ferrite material only in one direction of propagation. This kind of isolator works close to the resonance of the ferrite slab.

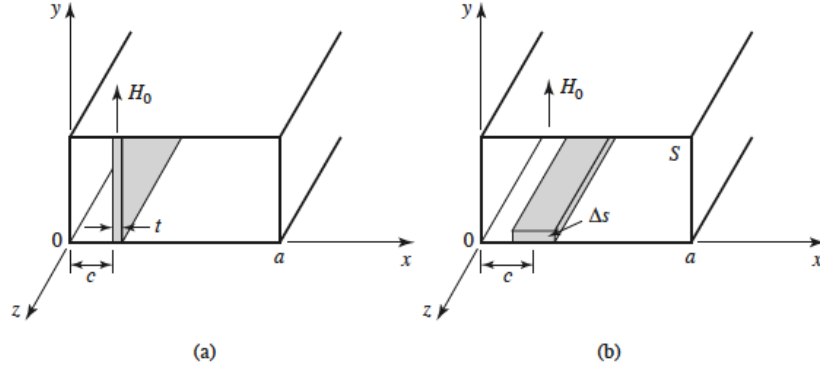


Figure 1.10 – Two resonance isolator configurations : (a) E -plane, full-height slab (b) H -plane slab. From [86].

The main application for the microwave isolator is to prevent possible reflections i.e. in an antenna or in a high power source.

1.3.2 Differential phase shifter

The differential phase shifter is a nonreciprocal two-ports device where the phase of the microwave signal has different velocities for forward and reverse signal, causing a controllable phase shift between both direction of propagation. The imaginary parts of the transmitted S -parameters have different values. The particular case of the microwave gyrator that have a 180° phase shift is represented by the scattering matrix

$$S = \begin{bmatrix} 0 & 1 \\ -1 & 0 \end{bmatrix}. \quad (1.44)$$

Many solutions allow inducing a switch to the phase of the signal, but only few solutions exist to induce a shift in the phase of both propagation directions. An example of nonreciprocal phase shifters is the ferrite toroid in a rectangular waveguide (Fig. 1.11), where a specific magnetization of the toroid sidewalls will interact differently with the forward or reverse rf fields [86].

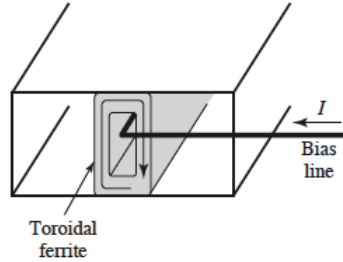


Figure 1.11 – Geometry of a nonreciprocal latching phase shifter using a ferrite toroid. From [86].

Applications for the microwave phase shifter are various. It can be used in test and measurements systems but its main application is in the phase array antennas where the beam can be electronically controlled by phase shifters.

1.3.3 Circulator

The microwave circulator is a nonreciprocal three-ports device that has the purpose to guide the signal between specific ports. The S -parameters can be written in a 3×3 -matrix

$$S = \begin{bmatrix} 0 & 0 & 1 \\ 1 & 0 & 0 \\ 0 & 1 & 0 \end{bmatrix}, \quad (1.45)$$

which shows the circulation of the signal power from port 1 to port 2, from port 2 to port 3 and from port 3 to port 1, but not in the reverse direction. Microwave circulators use biased ferrite substrates to control the signal flow (Fig. 1.12).

The nonreciprocity is using the counter-rotating modes in a cylindrical cavity. The biased ferrite material breaks the lowest-order resonant mode of the cavity into two resonant modes with slightly different resonant frequencies. The circulation occurs when the superposition of these two modes adds at the output port and cancels at the isolated port.

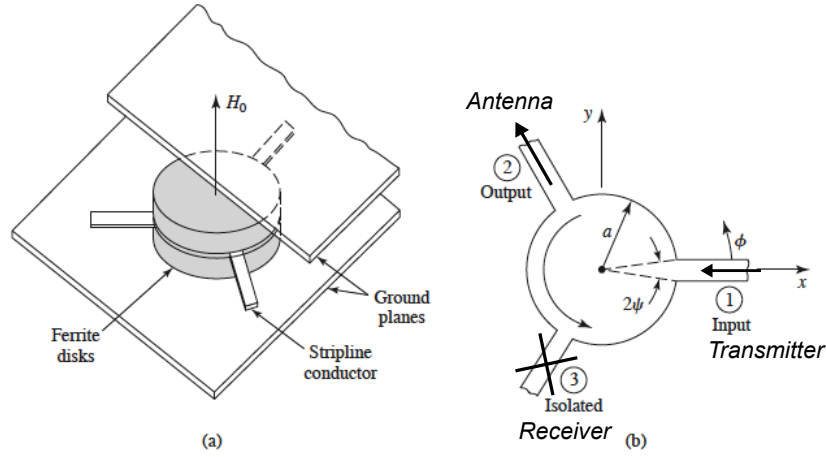


Figure 1.12 – A stripline junction circulator. (a) Pictorial view. (b) Geometry. From [86].

The three-ports junction topology can also be used as an isolator by terminating one of the ports with a short-circuited element.

The principal application of the microwave circulator is to guide the *rf* signal power in the circuit, i.e. from the transmitter to the antenna and from the antenna to the receiver.

1.3.4 Inductor

Microwave inductors are reciprocal devices used to enhance the inductance value of a circuit. To improve even more the inductance value, many inductors have a magnetic core in their structure with the important drawback that the ferromagnetic resonance and the eddy current induced in the volume of the core deteriorate the quality factor, which defined the ratio of its inductive reactance to its resistance, at *rf* frequencies. Micro- and nano-scaled topologies for the core structure were proposed to reduce these issues, as thin ferromagnetic layer, slotted or wire/strip patterned ferromagnetic films or micro/nano patterned ferromagnetic cores. At *rf* frequencies, the inductors use the FMR of the core structures that enhance their permeability for frequency range below the FMR frequency. Therefore, the working frequency range is below the FMR frequency.

Microwave inductors can be used as electronics filters, to separate the signals of different frequencies, or to block the *ac* signal while allowing *dc* to pass.

Chapter 2

Experimental techniques and basic *rf* characterization of nanowire arrays

2.1 Fabrication of ferromagnetic nanowired substrates

Several methods of fabrication are used to achieve a complete microwave device based on a ferromagnetic nanowire array embedded in a porous template. Parameters are easily tunable in all the process of fabrication, like the geometric parameters of the porous membrane or of the ferromagnetic nanowires. Configurations of transmission lines design that guides the electromagnetic signal into the nanowired substrate allow to develop series of microwave devices for which the *rf* properties can be measured thanks to different experimental techniques. Some characterizations of the geometric parameters or the intrinsic properties are also done at different steps of the fabrication. In this section, we will first explain the different kind of porous templates used for experimental measurements as well as their fabrication process and their tunable parameters. Then we will describe the electroplating step done in the purpose to obtain areas where nanowires grow inside the pores of membrane. A list of parameters will also be given for this fabrication step. A table resumes all the parameters of the samples used for the fabrication of microwave devices. We conclude this part with the explanation of the conception of the transmission lines design and their microwave measurements.

2.1.1 Electrodeposition into nanoporous templates

Porous polymer templates

Two kinds of porous polymer membrane are used in this thesis: the polycarbonate (PC) and polyvinylidene fluoride (PVDF) templates. Commercial polymer templates are provided by *it4ip*. Fabrication technique is the same for both kind of membrane [87]. A 25 μm -thick polymer film is exposed to high-energy ions bombardment perpendicular to its plane which will produce a track through the polymer chains (Fig. 2.1). A chemical treatment enlarges the pores to the desired diameter. Pores density or diameter are determined by the ions bombardment intensity and chemical etching time, respectively.

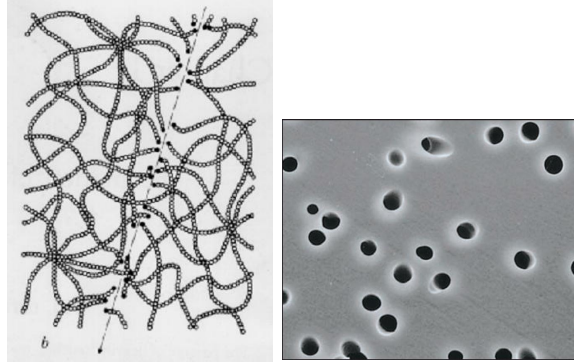


Figure 2.1 – Schematic track in polymer chains caused by motion of a high-energy ion creating the primitive pore and SEM image of the top of a PC porous membrane.

For the PC membrane, the thickness has an average of 21 μm , pore diameter from 29 nm up to more than 200 nm and the porosity from 1.3 % to 12 % are used. The simplicity and the low-cost of fabrication as well as a good dielectric constant ($\epsilon_r=3$) are the main features of the PC membranes. Straight pores and smooth pore walls complete its advantages. Unfortunately many drawbacks appear in the way to develop microwave applications with this kind of template: the first one is the poor thermal or chemical robustness, which avoids their uses in a standard circuit integration or in some fabrication techniques. In addition, inhomogeneous distribution of pores and deviations of their orientations may lead to significant uncertainties in the measured magnetic properties of nanowire arrays. For these reasons, PC membranes will serve in characterizations of magnetic properties to compare with nanowire arrays in PVDF membrane or porous alumina membrane.

PVDF membranes present the advantage to have piezoelectric properties due to the configuration of their carbons chains. The structure of the monomers chains of PVDF is $-(CH_2-CF_2)-_n$. The carbon-fluoride molecule ($-CF_2-$) presents a strong permanent electric moment due to the high electronegativity of the F atoms [88, 89]. β -phase (Fig. 2.2(b)) where molecule aligns its fluoride atoms on the one side of planar zigzag and all the hydrogen atoms to the other give the polarization of the chains.

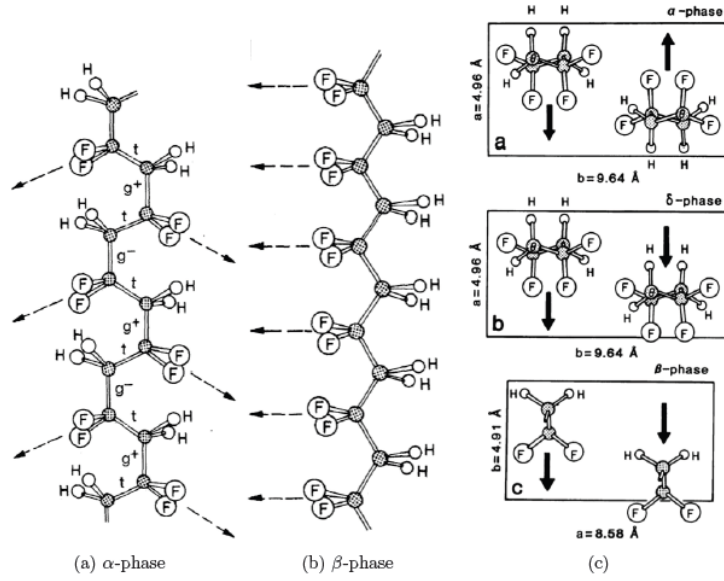


Figure 2.2 – Schematic structures in a side-view (a, b) and in a top-view (c) of a PVDF chain a) in a α -phase configuration and b) in a β -phase configuration. c) Schematic representation for the α -, β - and δ -phase respectively. The arrows represent the polarization. [89]

Piezoelectric properties will arise from this polarization. The strain ϵ resulting of the application of an dc electric field can be calculated for all the directions by

$$\epsilon_j = d_{ij} \cdot E_i, \quad (2.1)$$

with E_i the dc electric field and d_{ij} , the piezoelectric coefficients. $i = 1, 2, 3$ and $j = 1, 2, \dots, 6$.

Therefore, d_{33} induces strain in direction 3 per unit electric field in direction 3, d_{31} induces strain in direction 1 per unit electric field in direction 3 and d_{15} induces shear strain in direction 2 per unit electric field in direction 1. Fig. 2.3 shows all the axes directions.

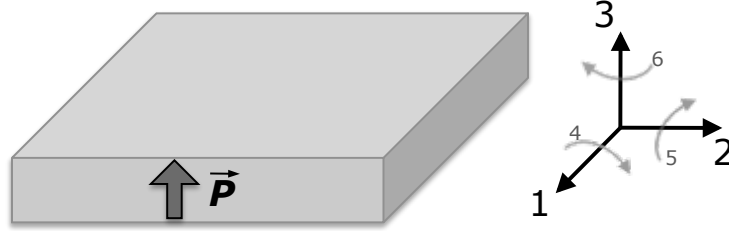


Figure 2.3 – Schematic structures of a PVDF template with electric polarization $\vec{P}$ perpendicular to the plane. Piezoelectric axes are defined.

The poling step is realized before the track-etching step and it consists to apply a large electric field (above 30 MV/m) that allows to enhance the piezoelectric coefficients, i.e. the perpendicular-to-the-plane coefficient d_{33} has its value increasing up to -35 pC/N [88]. A same process as the PC membranes will be used to obtain porous PVDF template. The thickness of PVDF membrane is 28 μm , the pore diameter from 105 nm up to 150 nm and the porosity from 1.7 % to 5 %. Thanks to the piezoelectric property of PVDF membrane, the conception of a new magnetoelectric nanocomposite is possible and it should provide a new route to control the magnetic properties of nanowire arrays under an electric field bias.

Porous alumina membrane

Self-supported porous alumina membranes are provided by *Synkera Technologies, Inc.* and by *Whatman*. Porous anodic alumina (AAO) membranes are fabricated in a two-step anodic oxidation process [90] from high purity Al foils or evaporated aluminum on Si substrates, depending on the required products (self-supported or Si supported AAO) (Fig. 2.4). A first anodization in oxalic acid solution leads to a hexagonal pattern on the surface of the Al foil. Then a second anodization step is carried out and pores through the hexagonal pattern will appear due to a reorganization of the atomic structures. A constant potential is applied through an acid solution during both anodizations. This potential, as well as the duration of each step or the pH of acid solutions, determine the pores interdistance or the height of the membrane. An additional chemical treatment is conducted to remove the remaining barrier layer and to enlarge the pores to the desired diameter.

The advantages of AAO membranes compared to porous polymer membranes are numerous in the development of microwave devices. First the good stability and robustness in temperature and under chemical

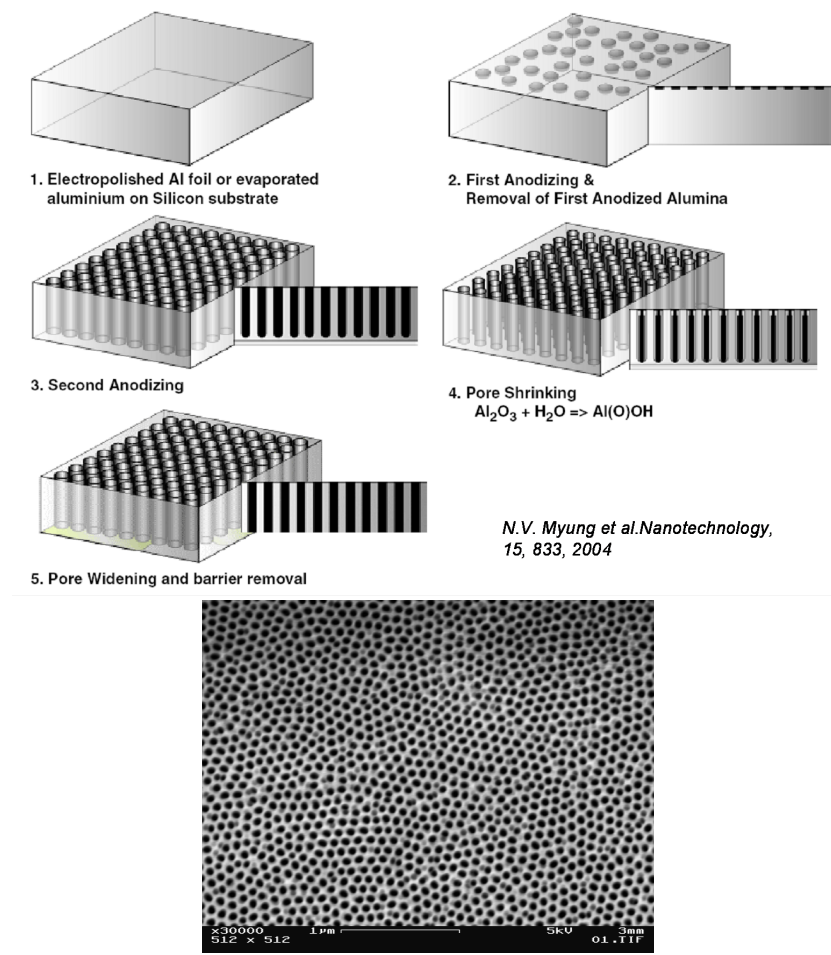


Figure 2.4 – Fabrication steps of porous anodic alumina membrane with the two anodization steps [90] and top-view SEM image of alumina membrane with pore diameter $\varphi = 60\text{nm}$.

products allow to develop some fabrication process to integrate this AAO membrane in microelectronic circuits. The good stability in the fabrication process leads to straight and smooth ordered nanopores and permits to obtain thick membranes and large porosities. A larger amount of magnetic material could be stored inside the AAO membrane, which enhance the response of magnetization to the microwave signal. The reproducibility is also improved thanks to the ordered hexagonal pattern. Dielectric properties of AAO membranes are also of interest to develop microwave devices. The high dielectric constant and low dielectric losses allow obtaining higher transmission.

Commercial AAO membranes from *Synkera Tech.* are used in the characterization experiments or in the microwave devices conceptions. The thickness of the membranes is 50 μm or 100 μm , the pore diameter are from 13 nm up to 150 nm and the porosity is ranging between 10 and 32 %. Commercial AAO membranes from *Whatman* are used for the fabrication of ferromagnetic inductors (chap. 5). This kind of membrane presents less reproducibility than the *Synkera* membrane. The average pore diameter in the used membrane lies in the range 100-150 nm, its porosity 25-50 % and the membrane height 60 μm . The dielectric constant of bulk alumina is $\varepsilon_b = 9.8$.

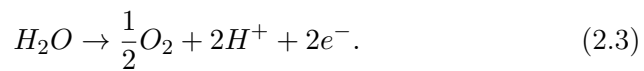
Electrodeposition

The electrodeposition is a bottom-up technique that consists in growing the metallic nanowires inside the pores of the template [23, 26]. It is an easy and cheap process of fabrication. Prior to the electrodeposition, metallic layers are deposited by *Pfeiffer 500* e-beam evaporator onto one face of the porous template to serve as the cathode electrode during electrodeposition. First a thin Cr layer is deposited on few nanometers thickness (5 to 20 nm) as an intermediate layer between PC/PVDF/alumina template and Cu/Au layer. Then, the gold layer is deposited with a thickness at least 4 times larger than the pore diameter to avoid leaks of the electrolytic solution. An additional Cu layer and Au layer are added to reach the thickness of 1 μm to reduce the ohmic losses of metallic layers due to the skin effect and protect the cathode from oxidation (Au).

Cells in Polytetrafluoroethylene (PTFE) polymer, called *Teflon*, are used to proceed to the electrodeposition. Rubber masks serve to determine the shape and surface of the electrodeposited area and to avoid any leakage. Electrolytic solutions made with ions from different metallic species are introduced into the cell and penetrate inside the nanopores of the membrane. A standard three-probe electrodeposition technique is used to induce a *dc* current through the solution and forces the cations to reduce on the cathode layer. The chemical reaction for single cation is:



where A refers to the metallic species of charge z and e^- to the electronic charge. At the opposite side, close to the anode electrode, occurs the oxidation reaction, which is the source of electrons and can be described as:



The deposited charge q can be quantified during the electrodeposition process by the following formula:

$$q = \int I dt, \quad (2.4)$$

representing the integral of electrodeposition current to the time. To determine the number of atoms deposited during the electrodeposition, we applied the following formula,

$$q = nMN_Ae^- = nMF, \quad (2.5)$$

where n is the number of electrodeposited molecules, M is the molar mass of the metallic element, N_A is the Avogadro number and F the Faraday constant. This calculation gives the number of deposited atoms in the hypothesis of a deposition efficacy of 100 %, which means that each reduce cation corresponds to a reduced electron.

For our samples, electrolytic solutions are based on metallic X compounds sulfates ($X - SO_4$). Boric acid is added to the solution to maintain its pH at constant value during the electrodeposition since different pH values influence the metal crystallinity [21]. The control of the solution pH can be made by adding H_2SO_4 or $NaOH$ respectively to decrease or to increase the pH. Two kinds of method can be used to induce a dc current through the solution: a galvanostatic method that applies a constant dc current to the electrodes or a potentiostatic method that applies a constant dc voltage. Only the second method has been used in our thesis. *EG&G Princeton Applied Research* potentiostat is linked to a computer that allows to observe the evolution of the electrodeposition current during the process. An $AgCl$ electrode, in addition to the anode and cathode electrodes, serves to control the voltage inside the solution (Fig. 2.5). Taking into account the reaction of the reference electrode $AgCl + e^- \rightarrow Ag + Cl^-$, The equilibrium voltage ($V = 0V$) compared to the neutral hydrogen reaction ($H_2 \rightarrow 2H^+ + 2e^-$) is situated to $+0.22$ V.

A dc constant voltage is applied through the solution by the potentiostat and the computer collects the information about electrodeposition current. During the process, the current value can be characterized by several stages symbolizing the evolution of the electrodeposition into the pores and outside the membrane. The first stage is the nucleation part of electrodeposition when nanowires start to grow at the bottom of the pores on the template cathode. This stage is symbolized by a maximum value at the beginning of the electrodeposition since the concentration of metallic ions is high. Then the second stage starts when all nucleations

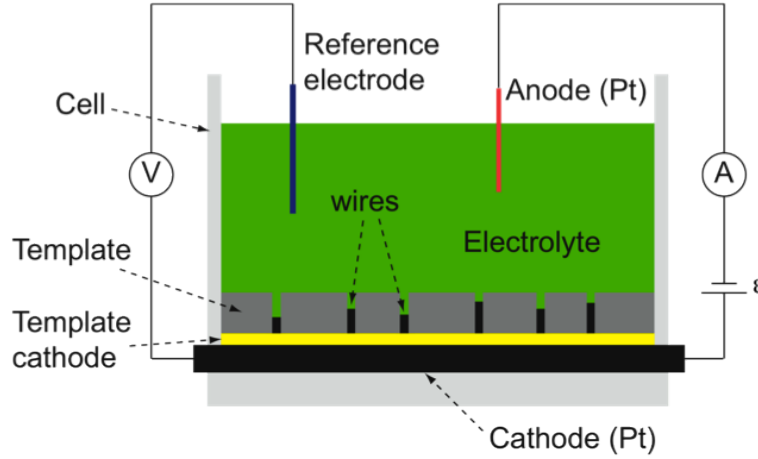


Figure 2.5 – Schematic representation of the electrodeposition cell with the three-probes configuration. The electrolyte enters inside the pores of the porous template until being in contact with the cathode. The *dc* voltage or current are controlled thanks to a potentiostat.

are made. A gradient of ions concentration takes place and the current is limited by the diffusion of ions into the pores. A relative constant current represents the 1D growth of nanowires. When apices of nanowires reach the top of the porous membrane, the electroplated material overflows the pores and can grow in 3D direction, which is represented by an increase of the current. After a time, the current increase is limited by the concentration of ions in the neighborhood of the electroplated material.

Electrolytic solutions used to grow the magnetic nanowires are listed in the table 2.1. The pH value of the solution is fixed after dissolution of items inside pure water by adding H_2SO_4 or $NaOH$ respectively to decrease or to increase the pH value.

In AAO membranes, due to the good reproducibility of their geometric parameters, nanowires growth is homogeneous and a fine control of the nanowires length can be done after a calibration step. Fig. 2.6 shows a side image from scanning electron microscope (SEM) of a NiFe nanowire array with homogeneous length ($45 \mu m$) embedded in a *Synkera* alumina membrane of height $90 \mu m$, pore diameter $35 nm$ and porosity 12 %. As we will see in section 2.2, the nanowires length is a key parameter that allows making microwave devices with tunable properties.

Solution	H_3BO_3	$NiSO_4$	$CoSO_4$	$FeSO_4$	Ascorbic acid	pH	E (V)
Ni	30	262.84	-	-	-	3.7	-1.1
Fe	45	-	-	120	1	3	-1.2
$Ni_{80}Fe_{20}$	24.73	131.42	-	5.56	-	3	-1.05
$Co_{55}Fe_{45}$	30	-	80	40	-	3.8	-0.9

Table 2.1 – Electrolytic solutions based on $NiSO_4 \cdot 6H_2O$; $CoSO_4 \cdot 7H_2O$; $FeSO_4 \cdot 7H_2O$ and boric acid (H_3BO_3). Unit of the compositions is gr/l.

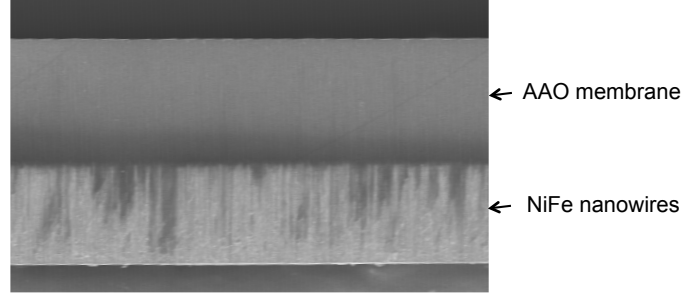


Figure 2.6 – SEM (scanning electron microscope) image from the side of a NiFe nanowire array embedded in a *Synkera* AAO membrane. The homogeneous length of nanowires is $h_w = 45 \mu m$ and the membrane height $h_p = 90 \mu m$. The pore diameter is $\varphi = 35 nm$ and porosity $P = 12\%$.

2.1.2 Configurations for microwave excitation

A transmission line is made on one side of the nanowired substrate to guide the microwave signal through the sample. Two kinds of configuration (Fig. 2.7) can be used: the stripline configuration where a transmission line is made on the other side of the cathode layer that will serve as ground layer in microwave measurements; the coplanar lines configuration where transmission line and ground planes are made on the same side of nanowired substrate. We forced the *ac* electric field resulting of the microwave signal to be between the transmission line and ground plane. The *ac* magnetic field takes place in the perpendicular direction to the *ac* electric field. Two fabrication methods are employed to make both configurations: a mask can be used to deposit the transmission line through, or a metallic layer can be etched to draw the design thanks to a laser micromachining system.

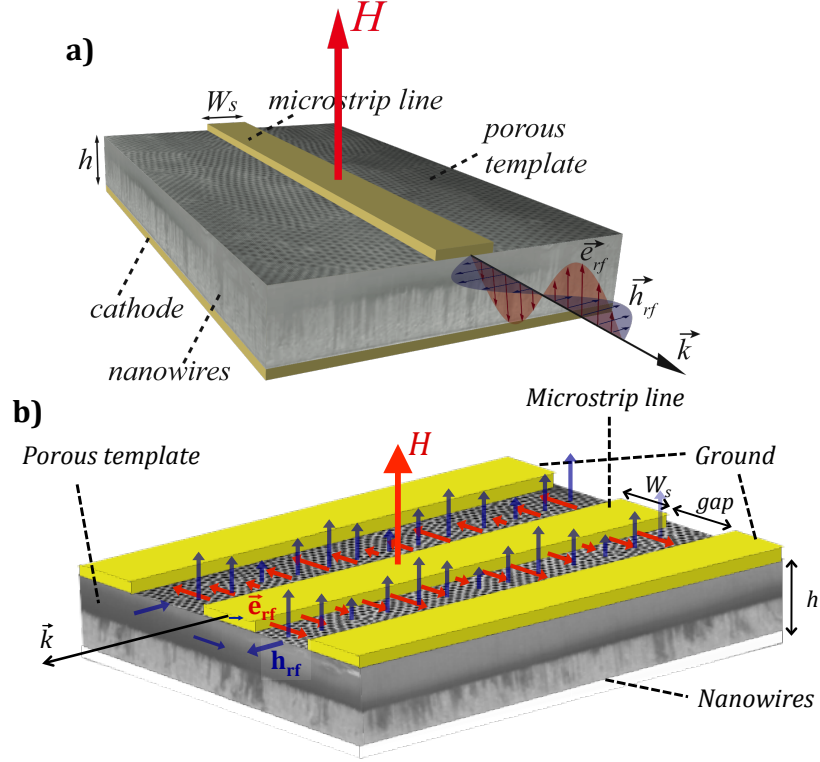


Figure 2.7 – Sketches of the transmission line in configurations (a) stripline and (b) coplanar. Nanowired sample is under a dc magnetic field H and a microwave of vector $\vec{k}$ signal passes through. Microwave electric $\vec{e}_{rf}$ and magnetic $\vec{h}_{rf}$ fields directions are indicated by red and blue arrows, respectively.

Stripline configuration

The transmission line and ground layer lay in the opposite side of the sample. In this configuration, the cathode layer used for electrodeposition can serve as ground. If the line width is much larger than its height, the energy of microwave signal is concentrated between the transmission line and the ground layer. The effects on the sides of transmission lines are confined and can be calculated thanks to the Hammerstad method [91]. The ac electric field is mainly concentrated inside the nanowired substrate and is parallel to the nanowires axis. The ac magnetic field is perpendicular to the ac electric field and is perpendicular to the nanowires axis, therefore to its magnetization when saturated magnetic states are considered. As we use Vector Network Analyzer (VNA) with a $50\ \Omega$ -internal impedance to inject the input microwave signal and analyze

the output signal, we should design the transmission line so that its impedance match with the one from VNA. A simple formula is used to calculate the line impedance compared to its width W_s [33]:

$$Z = \frac{377 h_p}{\sqrt{\varepsilon_R} W_s}, \quad (2.6)$$

where Z is the impedance, h_p is the membrane height, ε_R , the dielectric constant.

The first technique of making transmission lines uses a deposition method through a mask. Same *Pfeiffer 500* e-beam evaporator is used to deposit Cr (5-20 nm) / Cu (800nm) / Au (200 nm) layers. Two mask widths can be used for transmission lines, 150 μm and 500 μm . Typically, the 150 μm -wide transmission line matches better the 50 Ω -impedance value: for polymer membrane with $\varepsilon_R \sim 3$ and $h_p \sim 20\text{-}25 \mu\text{m}$, the line impedance has a value of about 30 Ω for the 150 μm wide transmission line while the impedance is about 9 Ω for the 500 μm wide transmission line. For AAO membranes, due to their higher dielectric constant $\varepsilon_R \sim 8$ (for $P \sim 12 \%$) and their thickness up to 50 μm or 100 μm , 150 μm is also the best width to match the transmission line impedance to 50 Ω . Although a narrow line is privileged to improve the transmission, wider lines (500 μm) are used in the case of low porosity membranes (around 1-2 % or even smaller) to probe the most amount of magnetic material. It allows to obtain enough absorption for the microwave signal.

The second technique to create a transmission line in stripline configuration consists to etch previously deposited metallic layer by a micro-machining laser system. Similarly to the first technique, Cr (5-20 nm) / Cu (800nm) / Au (200 nm) layers are deposited thanks to the *Pfeiffer 500* evaporator, but no mask are used. Metallic layers recover all the side of the sample. The etching of the metallic layer is done thanks to a 1064 nm laser with 5 % of its power $P = 13 \text{ W}$ and 50 passes to remove all the Cr/Cu/Au layers. A software program allow to draw design with fine precision. This method is time-consuming, only the specific designs of lines, as the circulator (Fig. 2.8) or coplanar lines, are done with this technique.

Coplanar lines configuration

The transmission line and ground layers lay on the same side of the sample. In this configuration, the cathode layer used for electrodeposition can either serve to be etched to design coplanar lines, or to be removed and in this last case, other metallic layer is deposited onto the other side of the sample where the transmission line will be etched. The

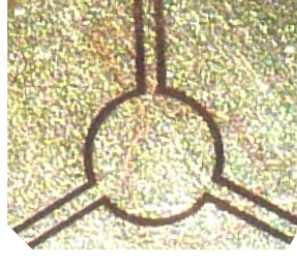


Figure 2.8 – Picture of a circulator based on NiFe nanowire array embedded in AAO membrane with $\varphi = 35$ nm, $h_P = 100$ μm and $P = 12$ %, which has been etched by the micromachining laser system. The device is in the microstrip configuration since the ground layer is deposited on the other side of the membrane. The remained gold layers, outside of the circulator design, are floating electrodes, therefore no *rf* field is expected to align with the coplanar configuration.

coplanar lines configuration allows to determine properties of the substrate thanks to the S-transmission matrix [92]. The coplanar lines can only be made by the etching of the micromachining laser system. Fig. 2.9 shows a test of coplanar lines etched on AAO membrane without any nanowires inside. The geometry of coplanar line is dependent of the used connectors, i.e. in Fig. 2.9 the pitch of the lines is 150 μm that can be connected with the appropriate connectors.

In the purpose to facilitate the lecture of the experimental results in chapters 3, 4 and 5 that discuss about microwave applications, all the fabricated samples are listed in table 2.2. For each sample, the kind of commercial alumina membrane (from *Synkera Tech* or *Whatman*) is indicated as well as the geometric parameters of the membrane (the pore diameter φ , the membrane height h_p and its porosity P). The magnetic materials used for each samples is written. The electrodeposition process for corresponding magnetic material as well as the concentration for NiFe and CoFe alloys are indicated in table 2.1. *Width* and *diameter* columns refers to the size of the designed transmission line. In chapter 3, the sample dimension only refers to the width of each zone. For chapter 4, the dimension refers to the diameter of the disc. The relative nanowires lengths, which are given in comparison to the membrane height, have been either measured in the scanning electron microscope (SEM) or calculated using the calibration method. The *lines fabrication*

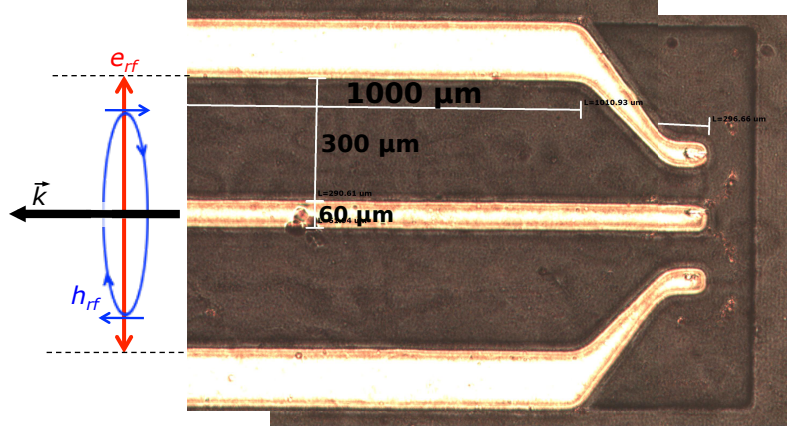


Figure 2.9 – Picture of etched coplanar lines on AAO membrane without nanowires. The characteristics of the AAO *Synkera* commercial membrane are: pore diameter $\varphi = 35$ nm, membrane height $h_p = 50$ μm and porosity $P = 12$ %. Coplanar lines are etched in metallic layers reported as Cr (5 nm) / Cu (800 nm) / Au (200 nm) and have a length 1 mm, entries length 300 μm , transmission line width 60 μm and a gap between transmission line and grounds 300 μm . Connections to the VNA can be done thanks to connectors with pitch 150 μm . No metallic layer is deposited in the opposite side of the membrane, therefore the *rf* electric and magnetic fields take place between the transmission line and the grounds.

column indicates the process used to design the transmission line (*mask*: deposited layer through mask; *laser*: etching of the design by the laser micromachining system; *lithography*: design of the line by the lithography etching process) and the number of ports in the case of nonreciprocal junction topology. The last column refers to the figures where results are presented for each sample. In the case of the ferromagnetic inductors, one may refers to the table 5.1 for a complete list of measured inductors.

2.1.3 Microwave measurements

A microwave signal is injected through the substrate by the transmission line (stripline or coplanar configurations) thanks to a Vector Network Analyzer (VNA) model *Anritsu 37397C*, which allows inducing microwave signal in the frequency range 40 MHz - 65 GHz. Both transmission amplitude and phase are measured and the signal power range is ± 10 dBm. The signal is suitable for microwave measurements like impedance, losses, isolation and transmitted and reflected signal. Several

Chapter 3 : Nonreciprocal devices based on microstrip line.									
#	Alumina membrane $\varphi(\text{nm})/h_p(\mu\text{m})/P(\%)$	Magnetic material		Width (mm)		Nanowires length (%)		Lines fabrication	Figure
		Zone 1	Zone 2	Zone 1	Zone 2	Zone 1	Zone 2		
1	<i>Synkera</i> 35/100/12	CoFe	NiFe	4	0.5	68	84	<i>Mask</i>	Fig. 3.3
Chapter 4 : Nonreciprocal three-ports junction topology.									
#	Alumina membrane $\varphi(\text{nm})/h_p(\mu\text{m})/P(\%)$	Magnetic material	Diameter (mm)	Nanowires length (%)		Lines fabrication	Figure		
1	<i>Synkera</i>	NiFe	3	40	3-ports - <i>Laser</i>		Fig. 4.4 Fig. 4.5(a)		
2				50					
3	35/100/12			56					
4				60					
5	<i>Synkera</i>	CoFe	4	50			No fig.		
6	35/100/12			60					
7	<i>Synkera</i> - 18/50/10			62					
8	<i>Synkera</i>	NiFe	3	50	2-ports - <i>Laser</i>		Fig. 4.9		
9				56					
10	35/100/12			60					
11	<i>Synkera</i>	CoFe		65			Fig. 4.9		
12	18/50/10			60					
Chapter 5: Ferromagnetic inductors.									
#	Alumina membrane $\varphi(\text{nm})/h_p(\mu\text{m})/P(\%)$	Magnetic material	Nanowires length (%)		Lines fabrication	Figure			
1	<i>Whatman</i> - 100/60/35	Ni	40		<i>Lithography</i>	see tab. 5.1			

Table 2.2 – Parameters of the fabrication for all measured samples used in the characterization of the microwave devices in chapter 3, 4 and 5 and links to the figures showing the corresponding experimental results.

kinds of method are used to record the microwave measurements.

Frequency spectra measurements

Frequency spectra are recorded for frequency range 40 MHz - 65 GHz for the signal passing through the Device Under Test (DUT). This DUT can be a non-magnetized sample (porous membrane without ferromagnetic nanowires) as well as magnetic nanowired sample. In the last case, a saturation of the magnetization up to 10 kOe is done before the measurements by placing the DUT inside the core of electromagnet that is supplied with a FUG voltage power (40 A - 240 V) source model *NTN 10400M - 260* (Fig. 2.10). The Hall sensor inserted in the core of electromagnet and supplied by a 100 mA current source allows controlling precisely the magnetic field. *Labview* software on computer is used to transmit all the commands and to receive information by GPIB cables. The VNA injects an input microwave signal power P_{in} through the DUT and retrieves an output microwave signal power P_{out} . We collect the S-transmission matrix with amplitude (dB) and phase (rad). Before the measurements, the VNA is calibrated by the Short-Open-Load-Through (SOLT) method using coaxial calibration kit. Recording frequency spectra at constant applied magnetic field can also be done.

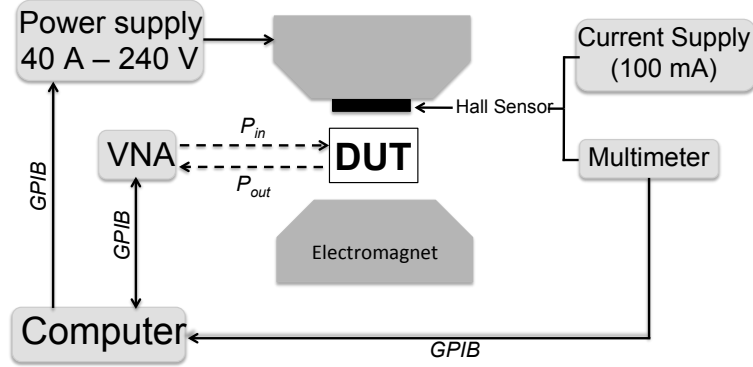


Figure 2.10 – Schematic representation of the measurement setup. The Device Under Test (DUT) is placed in the core of the electromagnet and linked to Vector Network Analyzer (VNA). A *dc* magnetic field is applied thanks to a voltage power supply and controlled with a Hall sensor. The VNA injects an input microwave signal power P_{in} through the DUT and analyze the output microwave signal P_{out} . All the operations are controlled by Labview software installed in the computer.

Results are given in the form of S-transmission matrix where trans-

mission is recorded in both directions. S-matrix gives precise information about the conductive and dielectric losses of the substrate, as well the FMR absorption spectra if magnetic nanowires are included in the sample. In the case that we consider only the behavior of the FMR absorption spectra, correction on the transmission frequency spectra can be done by subtracting the high magnetic field transmission spectra from the transmission. The remaining information is then only about the FMR absorption.

Magnetic field spectra measurements

Thanks to a fine control of applied magnetic field on the DUT, magnetic field sweep measurements in the range 0 - 10 kOe and at constant frequency in the range 40 MHz - 65 GHz can be recorded. This kind of measurement is used to obtain fine information about the FMR absorption without considering the other conductive and dielectric contributions. It is really important in the case of small amount of magnetic material embedded in high-losses dielectric templates. Magnetic field sweep permits to probe weak FMR absorption. Spectra of magnetic field sweep at different constant frequencies give information about the resonant magnetic fields that can be reported on the dispersion relation of the magnetic sample (Fig. 2.11). This dispersion relation depends on the gyromagnetic ratio γ of the material and it can be calculated knowing all the anisotropy contributions of the magnetic nanowired substrate. In the case of the only contribution is the shape anisotropy, the dispersion relation can easily be found by the formula [23]:

$$f_{FMR} = \gamma (H_{app} + 2\pi M_s(1 - 3P)). \quad (2.7)$$

Low temperature measurements

A probe station measurements setup from *Desert Cryogenics* is used to cool the DUT to the liquid nitrogen temperature (77 K). Samples are placed inside the probe station where vacuum is made, and is connected to the VNA thanks to coaxial access connections. A *Lakeshore 331* Temperature Controller connected to the chamber measures the temperature of the sample thanks to a Cernox sensor and allows to obtain any temperature between 77 K and 325 K using a heating resistor. Both frequency and magnetic field sweeps are recorded in this kind of measurements since the probe station is placed inside the core of electromagnet. Low temperature measurements will be carried out to analyze the magnetoelastic effect that appears when cooling ferromagnetic nanowire arrays in polymer membrane and also to understand the behavior of unexpected contribution to the magnetic anisotropy.

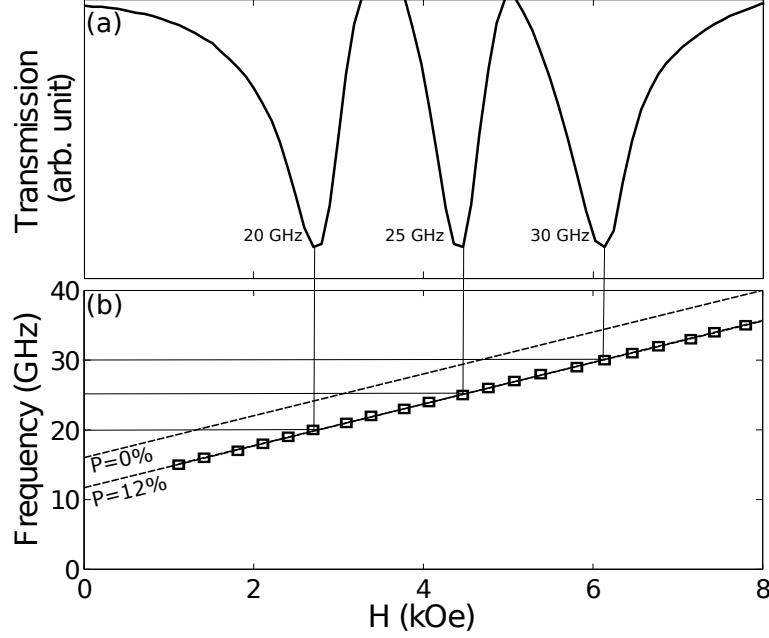


Figure 2.11 – (a) Magnetic field sweep for a NiFe nanowire arrays embedded in a commercial AAO membrane with pore diameter $\varphi = 35$ nm, membrane height $h_p = 50$ μm and porosity $P = 12\%$ for three different constant frequencies. FMR absorption peak is reported in (b) showing the dispersion relation of the sample. Dashed lines are calculated thanks to the model of magnetic anisotropy for $P = 0\%$ and $P = 12\%$. The slope of the dispersion relation is equivalent to the gyromagnetic factor $\gamma = 3 \text{ GHz}\cdot\text{kOe}^{-1}$.

Temperature and humidity chamber measurements

Once again, this method permits to carry out microwave measurements at different temperatures but also to control the humidity of environment (Fig. 2.12). We use a Temperature and Humidity Chamber model *Espec SH-261* to control the temperature in the range -60°C to 150°C and the humidity in the range $30\% \text{rh}$ - $95\% \text{rh}$. Measurements are done on saturated magnetic substrates since no external magnetic field can be applied at the same time. Only the frequency sweep can be performed using this method. The dependence of magnetic and microwave properties of nanowired substrates is studied here to prove the good stability of microwave devices based on nanowire arrays with respect to the temperature variation.

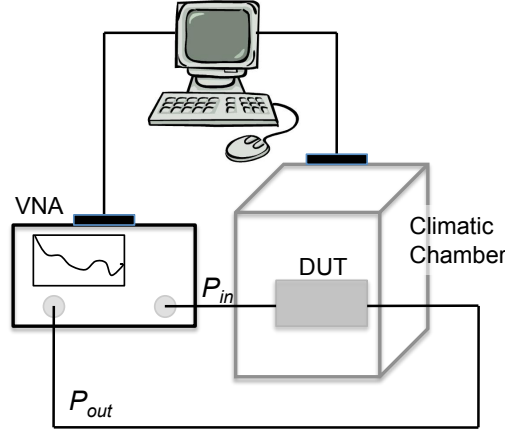


Figure 2.12 – Schematic representation of experimental measurements on DUT in the climatic chamber.

Coplanar lines measurements

Coplanar lines measurements can be done on the adapted DUT inside the core of electromagnet or can be performed on a prober model *SuSS PM8* with adapted $150\ \mu\text{m}$ -pitch Ground-Signal-Ground (GSG) coplanar probes. In this last case, the calibration of VNA is done using an Impedance Standard Substrate that avoids the mismatching of the sample holder. The probe station is linked to *Anritsu* VNA with frequency range 40 MHz - 40 GHz. Frequency sweeps are performed on coplanar lines with pitch of $150\ \mu\text{m}$.

2.2 Basic *rf* characterization of ferromagnetic nanowire arrays

An important advantage of ferromagnetic nanowire arrays is the control of its intrinsic magnetic and dielectric properties thanks to some geometric considerations. Indeed, a ferromagnetic nanowires array embedded in porous polymer or alumina membrane can be characterized by its effective permeability or its effective permittivity. Theoretical models have been developed to predict the behavior of these parameters at microwaves. The effective permeability controls the ferromagnetic resonance of the nanowire array with dependences including the nature and the volume (porosity P and nanowires length h_w) of the magnetic material. In addition to the effective permeability, the effective permittivity can be easily controlled thanks to a theoretical model developed by J.

Spiegel [35] in 2007, which obtained good agreements with experimental results. Both magnetic and dielectric properties allow to conceive and to design microwave devices but also to tune their microwave behavior.

On the other hand, losses are quite important issues in the microwave applications to assure low insertion losses as usually required in industrial specifications. Dielectric losses are exclusively dependent on the effective permittivity of the membrane since the presence of metallic nanowires does not act on the loss tangent factor [35]. We measured relatively high dielectric losses in the commercial AAO membranes that we used to conceive microwave applications, which has the consequence to present high insertion losses. Then we investigated several fabrication methods or crystallographic structural comparisons in the purpose of reducing these dielectric losses. A characterization of conductive losses from the measurement of transmission lines has been realized in a similar goal.

2.2.1 Effective permeability

The equation of high-frequency permeability is written in the section 1.2.3 using the high-frequency susceptibility calculated in the case of small damping thanks to the equation of magnetization motion. The determination of permeability tensor components has to take into account the geometric parameters of nanowire array as the porosity P or the nanowires length h_w , as well as the normalized remanent magnetization $m = M/M_s$.

The effective permeability tensor of ferromagnetic nanowire array can be synthesized by the formula:

$$\bar{\bar{\mu}}_{eff} = \begin{bmatrix} \mu & j\kappa & 0 \\ -j\kappa & \mu & 0 \\ 0 & 0 & 1 \end{bmatrix}, \quad (2.8)$$

with

$$\begin{aligned} \mu &= 1 + 2\pi M_s \gamma P h_w f_r (f_r^2 - f^2)^{-1} (m^2 + 1), \\ \kappa &= 4\pi M_s \gamma P h_w f (f_r^2 - f^2)^{-1} m, \end{aligned} \quad (2.9)$$

where γ is the gyromagnetic factor, M_s the magnetization saturation and $f_r = \gamma(H + H_{eff})$ the FMR frequency.

The non-diagonal terms κ of the tensor can be seen as the non-reciprocal factor of the nanowired substrate. A high normalized remanent magnetization m , but also a large magnetic volume (porosity P and nanowires length h_w) are needed to obtain large non-reciprocal effects in microwave device. In this purpose, geometric parameters of the nanowired substrate

will be chosen to promote high remanence (low porosity P) with high amount of magnetic matter (large nanowires length h_w). This model permits to know the value of the effective permeability in high-frequency range and to include it in numerical simulations, which give the microwave behavior.

As explained in [34], experimental measurements of the effective permeability from nanowired substrate can be achieved with the method of transmission lines. The S -(transmission and reflection) matrix is transformed into a T -chain matrix. Both effective permittivity and permeability can be calculated thanks to the S -matrix. Fig. 2.13 shows the real and imaginary parts of the effective permeability measured thanks to the transmission line method for a NiFe nanowire array embedded in a AAO membrane with $\varphi = 60$ nm, $P = 25$ %, $h_p = 60$ μ m and $h_w = 55$ %. An external dc magnetic field is applied parallel to the nanowires to saturate the sample. A mismatch between the real impedance of the sample on its holder and the 50 Ω -impedance of the VNA induces small ripples in Fig. 2.13.

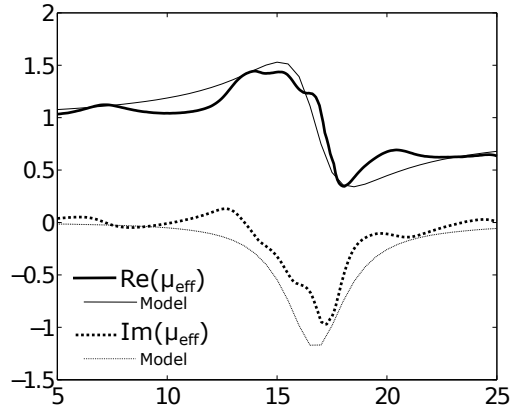


Figure 2.13 – Real (solid lines) and imaginary (dotted lines) parts of the effective permeability measured for NiFe nanowire arrays embedded in an AAO membrane with $\varphi = 60$ nm, $P = 25$ %, $h_p = 60$ μ m and $h_w = 55$ % and with external magnetic field $H = 5$ kOe. Experimental results (thick lines) have good agreement with the model (thin lines).

The tunability of the effective permeability on the ferromagnetic material of nanowire arrays will be used to conceive planar microwave inductors (see chapter 5). The presence of magnetic nanowires in the substrate of inductors will improve the value of inductance without increasing the

losses of the system. We will also develop a microwave isolator and differential phase shifter by including different magnetic matters in two areas below the transmission line (see chapter 3). Thanks to the difference in the intrinsic properties of both zones, microwave signal will flow differently in the two directions of propagation. Finally the microwave circulator will have a dependence on the effective permeability for which circulation effects will take place below and above the FMR frequency (see chapter 4).

2.2.2 Effective permittivity

As demonstrated and experimentally proved in a previous paper [35], the effective permittivity of a nanowired substrate depends on the length (h_w) and the porosity (P) of the nanowire array. Basically, using a quasi-static model taking into accounts only the fundamental mode, the capacitance of a nanowired substrate can be considered as a two-layers system divided as nanowired part and air gap. Fig. 2.14(a) and (b) shows the side view schematic representation of nanowire array and its corresponding equivalent circuit for the line capacitance. This model is valid for a microstrip configuration with thin layer substrate, the rf electric field direction parallel to the wire axis and for nanowires with diameter much smaller than skin depth.

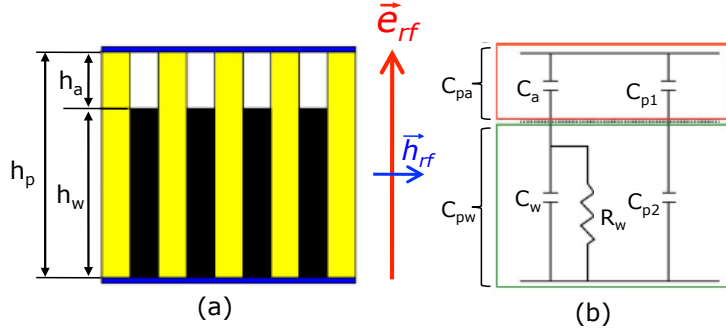


Figure 2.14 – (a) Side view schematic representation of nanowired substrate with membrane height h_p , nanowires length h_w and gap $h_a = h_p - h_w$. (b) Corresponding equivalent circuit for the line capacitance for the two-layers model with the nanowired part (green area) and air part (red area). Blue and red arrows are the direction of the rf electric and magnetic fields. [35]

As the partial capacitances are in parallel, we can calculate the ca-

capacitance of each layer of the system

$$\begin{aligned} C_{pa} &= C_a + C_{p1}, \\ C_{pw} &= C_w + C_{p2}. \end{aligned} \quad (2.10)$$

Then the total capacitance of the nanowired substrate results from the connection in series of C_{pa} and C_{pw}

$$C_{tot} = \frac{C_{pa}C_{pw}}{C_{pa} + C_{pw}}. \quad (2.11)$$

The formula of the effective permittivity of nanowired substrates is written as

$$\varepsilon_{eff} = h_p \left(\frac{h_w}{\varepsilon_w} + \frac{h_a}{\varepsilon_p} \right)^{-1}, \quad (2.12)$$

where h_p is the height of the membrane, h_w the nanowires length, h_a the air gap between the top of nanowires and the surface membrane, ε_p and ε_w the permittivity for the air gap and the nanowired part defined as

$$\begin{aligned} \varepsilon_p &= P + (1 - P) \varepsilon_b (1 - j \tan \delta_b), \\ \varepsilon_w &= P \left(1 - j \frac{\sigma_w}{\omega \varepsilon_0} \right) + (1 - P) \varepsilon_b (1 - j \tan \delta_b), \end{aligned} \quad (2.13)$$

where P is the porosity, σ_w the conductivity of the metallic nanowires, $\tan \delta_b$ the dielectric loss tangent of bulk membrane and ε_b its dielectric constant.

Thanks to this theoretical model, knowing the geometric parameters of nanowired substrate will allow to calculate the value of the effective permittivity of a nanowired substrate. Fig. 2.15 represents a numerical calculation of the real part of the effective permittivity of a NiFe nanowire array embedded in AAO membrane with $\varepsilon_b = 9.8$, $h_p = 60 \mu\text{m}$, $\sigma_w = 5.9 \cdot 10^7 \text{ S/m}$ and $\tan \delta = 6.7 \cdot 10^{-2}$, and depending on the relative nanowires length h_w and on the porosity P .

Experimental results have shown good agreements with the model [35] for membranes with low porosity ($P = 2 \%$). The validity of the model can be extended to larger porosities up to $P = 25 \%$ since the nanowires diameter remains smaller than skin depth in metallic nanowires to avoid conductive losses. This porosity range includes all the microwave devices designs where calculations of ε_{eff} will be needed. The imaginary part of the effective permittivity evolves in the same proportion as the real part, with the consequence that the dielectric loss $\tan \delta$ of the substrate does not increase in presence of metallic nanowires. Low dielectric losses are crucial to ensure a good transmission of

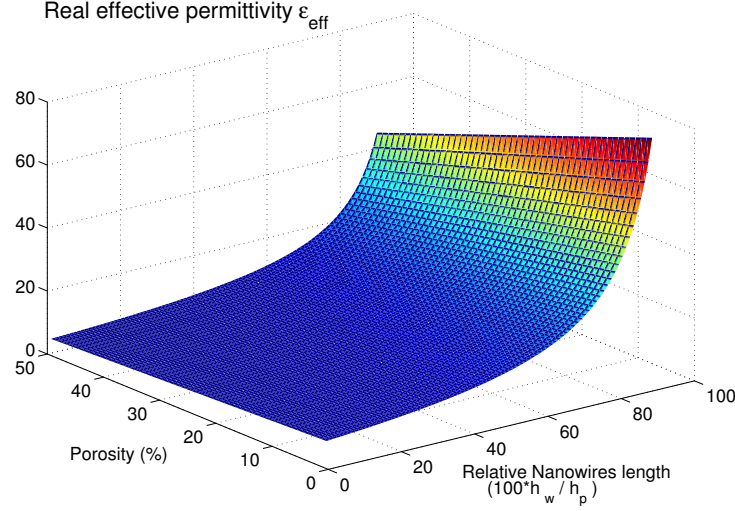


Figure 2.15 – Calculated real part of effective permittivity of a NiFe nanowire array embedded in an AAO membrane with $\varepsilon_b = 9.8$, $h_p = 60 \mu\text{m}$, $\sigma_w = 5.9 \cdot 10^7 \text{ S/m}$ and $\tan \delta = 6.7 \cdot 10^{-2}$, and depending on the relative nanowires length h_w and on the porosity P .

microwave applications.

The determination of effective permittivity value will be important to conceive microwave circulators for which the circulation frequency has a fine tunability to this intrinsic value (see chapter 4). The control of the nanowires length will permit to tune finely the working frequency of the device. Microwave differential phase shifters will also have a dependence on the effective permittivity that controls the value of the phase shift (see chapter 3).

2.2.3 Dielectric losses in substrate materials

Insertion losses of microwave devices based on nanowire arrays embedded into porous membranes are the main issues on the way to an industrial transfer. Companies active in microwave technologies have some required specifications for the insertion losses, which vary depending the applications and working frequencies. We defined the insertion losses as the loss in a transmitted signal passing through a substrate. The unit is written in logarithmic scale: $L = -20 \log_{10} |S_{21}|$. Several causes may intervene in the determination of the global insertion losses

in a nanowired substrate, as the dielectric losses of the porous matrix or the ohmic losses of the signal and ground metallic lines. As expected, the presence of metallic nanowires of small diameter does not increase the dielectric losses if their diameter is smaller than the skin depth over all analyzed frequencies range. Fig. 2.16 shows the transmission S_{21} of a stripline with a width $150\ \mu\text{m}$ and a thickness $1\ \mu\text{m}$ deposited on a commercial *Synkera* AAO membrane. Dashed lines are a guide for the eyes and identify the respective contributions. As we can see, ohmic losses are rapidly saturated while increasing the frequency. Dielectric losses depend on the frequency and increase all along the frequencies spectra.

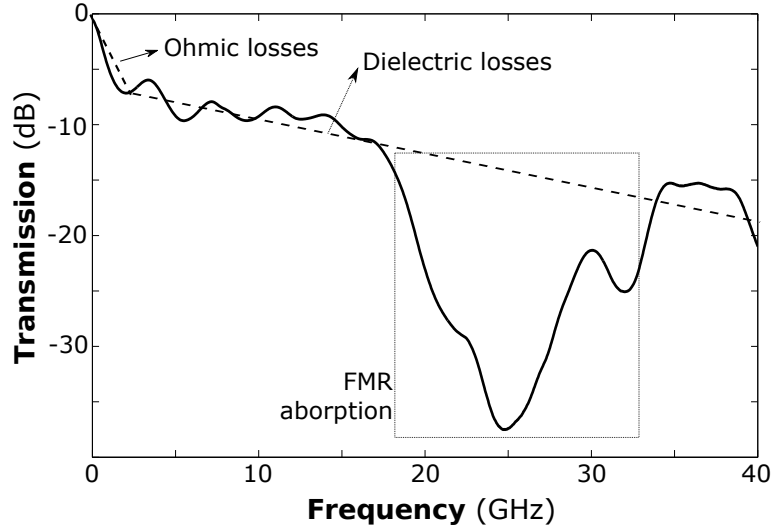


Figure 2.16 – Transmission spectra for a stripline with width $150\ \mu\text{m}$ and thickness $1\ \mu\text{m}$ deposited on a commercial *Synkera* AAO membrane including Fe nanowires with pore diameter $\varphi = 35\ \text{nm}$, membrane height $h_p = 100\ \mu\text{m}$ and porosity $P = 12\ \%$. Dashed lines are guide for the eyes to symbolize the ohmic and dielectric losses of the samples.

High insertion losses have been observed in the microwave devices fabricated and characterized in this thesis, for example in the differential phase shifter (chap. 3) or in the microwave circulator (chap. 4). Despite of the high non-reciprocal effects observed in these devices, the measured insertion losses remain prohibitive to compete with the commercially available ferrites devices. A deep study is necessary to reduce the sources of high losses by searching to improve the quality of the nanowired substrate and transmission line, or to develop composites with relatively-low dielectric materials.

Dielectric losses of AAO membrane

Dielectric losses of nanowired substrates is synthesized in the loss tangent factor using the formula

$$\tan \delta = -\frac{\varepsilon''}{\varepsilon'}, \quad (2.14)$$

where $\varepsilon_{eff} = \varepsilon' - j\varepsilon''$ is the effective permittivity of the nanowired substrate that can be calculated using the formula (2.12). We know that real and imaginary parts vary in the same proportion when including nanowires inside the membrane, so $\tan \delta$ does not increase in the presence of nanowires. The variation of the total dielectric losses can be seen in the dependence of ε_{eff} to the dielectric losses of bulk template $\tan \delta_b$.

We only treated the dielectric losses in AAO membranes since the polymer porous templates are not used for the design of microwave applications in this thesis. Insertion losses in the measurements of differential phase shifters or microwave circulators are the consequence of high dielectric losses of the commercial AAO membranes used for their fabrication. We estimated from a correspondence obtained between experimental and numerical results that the value of dielectric losses in this kind of AAO membrane is in the range 0.02 to 0.07. It should be noted that thin layers of alumina material are used as microwave dielectric substrates, with a reported nominal loss tangent factor as low as $\tan \delta = 10^{-4}$ [93]. Besides the porosity of the membrane is expected to influence its dielectric losses [93]. For a porosity of 12 %, we considered that an attainable value of dielectric losses, in a case of dry membrane [94], can be 10^{-3} . Due to the porosity of the membrane, the atmosphere during the measurements or during the fabrication is linked to the variation of the dielectric losses. We will first study the influence of an adapted fabrication of nanowired substrates to remove the humidity of the sample by drying and covering it with a Si_3N_4 protection layer. We will then characterize the commercial *Synkera* AAO membrane with their crystallographic structural organization obtained by thermal treatments to observe the difference of dielectric properties.

Influence of humidity on porous alumina

The fabrication of nanowired substrates using liquid solution is favorable for the residual moisture in the pores of the membrane. To avoid the presence of H_2O molecules inside the pores, we will dry the nanowired substrate by heating it to 350 °C during 4h in a vacuum chamber and simultaneously, we will cover the top of the substrate with a protective Si_3N_4 layer by PECVD techniques. This protective layer has a 200 nm-thickness, which is enough to cover all the pores. We use NiFe

nanowire arrays deposited in the same AAO membrane and with the same electrodeposition parameters to compare the results. A $150\ \mu\text{m}$ -wide, $1\ \mu\text{m}$ -thick and $1\ \text{cm}$ -long stripline is deposited on each nanowired substrate.

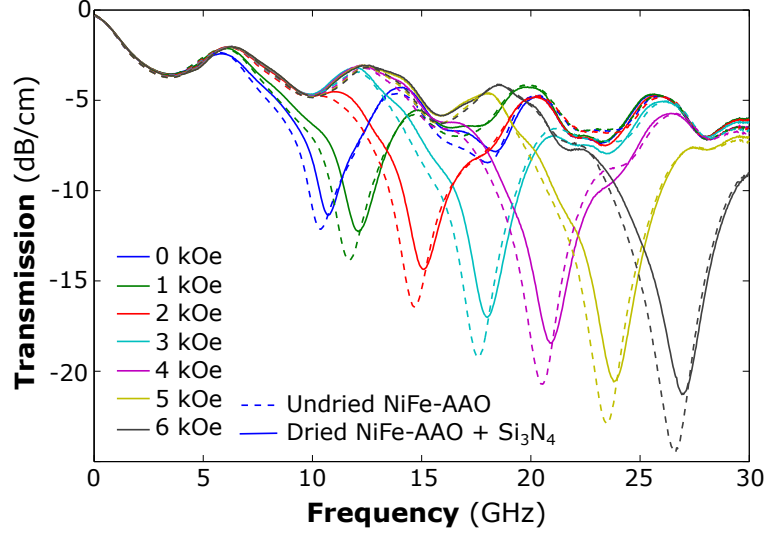


Figure 2.17 – Transmission spectra of similar NiFe nanowired substrates in the same AAO membrane ($\varphi = 35\ \text{nm}$, $h_p = 50\ \mu\text{m}$ and $P = 12\ \%$) for different applied magnetic fields. An additional step is added in the fabrication: we dry the nanowired sample in vacuum chamber at $350\ ^\circ\text{C}$ during 4h and we cover it by a protective Si_3N_4 layer by PECVD. Solid lines represent the dried and covered sample, dashed lines the non-dried and uncovered sample.

In Fig. 2.17, we can observe the transmission spectra between the non-dried and uncovered NiFe nanowire array (dashed lines) and the dried and covered NiFe nanowire array (solid line) at different applied magnetic field. Peaks are the FMR absorption and the FMR frequency is located near $10\ \text{GHz}$ for $0\ \text{kOe}$ magnetic field. Excepted a small shift in the FMR frequency and amplitude in the case of the dried and covered sample, which may result on an oxidation of NiFe to NiFeO_4 and then a smaller porosity of the array, no difference is observed in the insertion losses between both samples.

Other samples with a variation of the nanowires length confirm these results. It could mean that either no enough drying time or high temperature is applied to remove all the H_2O molecules from pores, the

humidity would remain in both samples, or depositing the transmission line would also dry the nanowired substrate, the humidity would be removed from both samples.

Comparison on different crystallographic structures of AAO membranes

Commercial *Synkera* AAO membranes have an amorphous crystallographic structure due to their fabrication process. Heat treatments can change the crystallographic structure from amorphous to γ - or α -alumina and allow to increase the maximum operating temperature. In order to compare the performance of each kind of untreated and treated AAO membranes, we bought alumina membranes having the same parameters (pore diameter $\varphi = 35$ nm, membrane height $h_p = 50$ μm and porosity $P = 12$ %) but with the different heat treatments (table 2.3).

Heat treatment	Max. operating temperature	Phase
No heat treatment	500 °C	amorphous
#1	700 °C	amorphous
#2	900 °C	γ -alumina
#3	1000 °C	γ/α -alumina

Table 2.3 – Heat treatments for commercial *Synkera* AAO membranes with their maximum operating temperatures and the crystallographic phases.

Dielectric properties of the AAO membranes will be studied without the presence of nanowires. A metallic Cu/Au layer of thickness 1 μm is deposited in one side of the membranes and coplanar lines structures with various lengths (1 mm, 2 mm and 4 mm) are etched using the laser micromachining system (Fig. 2.18). Previous numerical simulations gave the geometric parameters of coplanar lines to match the 50 Ω -impedance of the VNA. The transmission line width is 60 μm and gaps between the transmission line and grounds are 30 μm . Fig. 2.18 is the schematic representation in the software AlphaCAM used to draw the design etched by the laser (in cyan). The inset is a picture of etched coplanar lines.

Measurements are done on the prober SuSS PM8 with 150 μm -pitch coplanar probes and a VNA working in the frequencies range 40 MHz - 40 GHz. Fig. 2.19 shows the comparison in the transmission amplitude between all kinds of alumina membranes. We observe that the amorphous alumina structures (solid and dotted curves) have better transmissions than γ - or γ/α -crystallographic alumina membranes (short- and long-dashed curves).

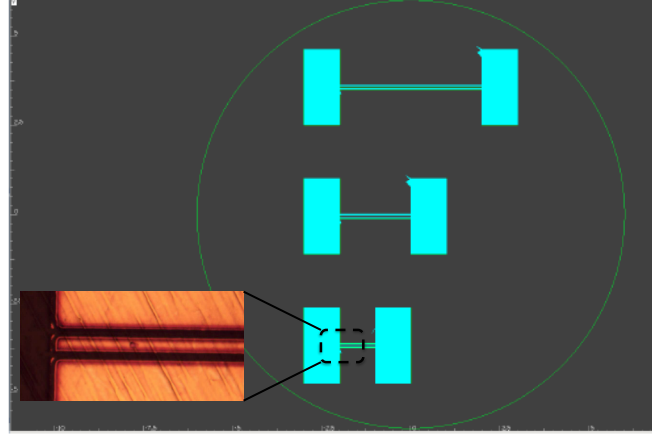


Figure 2.18 – Schematic design of coplanar lines drawn in AlphaCAM software to proceed to the etching of the Cu/Au layer by the laser micromachining system. Cyan areas are the etched part. In inset, picture of coplanar lines after the etching.

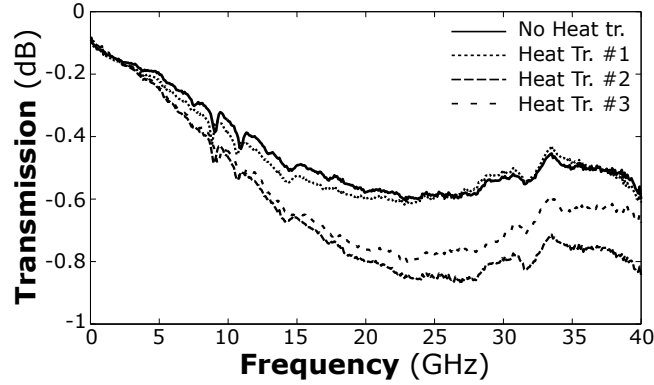


Figure 2.19 – Transmission spectra for 2 mm-long coplanar lines of all commercial *Synkera* AAO membranes with different heat treatments. Solid and dotted lines have an amorphous crystallographic structure, short- and long-dashed lines have respectively γ - and γ/α - alumina structures.

For each sample, we can extract the dielectric properties thanks to the technique [92] using the S -matrix for two different lengths of coplanar lines. It permits to remove the mismatch of the coplanar line compared to the $50\ \Omega$ -impedance. Fig. 2.20 shows the value of the dielectric constant ($\text{Re}(\epsilon_r)$), the imaginary part of the permittivity ($\text{Imag}(\epsilon_r)$) and the loss

tangent factor ($\tan \delta$) for each kind of *Synkera* AAO membranes (No heat treatment, Heat treatments #1, #2, #3) depending on the frequency. The first general observation is that the procedure used to extract the dielectric properties is not valid at the low frequencies range (< 5 GHz) because it does not take into account the conductive losses that are predominant at these frequencies. Secondly, the measured values of the dielectric constant ε_p can not be related to the calculated values using eq. (2.12), since the configuration of the *rf* electric field is not perpendicular to the sample plane. If we compared the loss tangent factor, $\tan \delta$ of samples without and with #1 heat treatment have lower values than samples with #2 and #3 heat treatments in all the frequencies range, which confirms the results in Fig. 2.19.

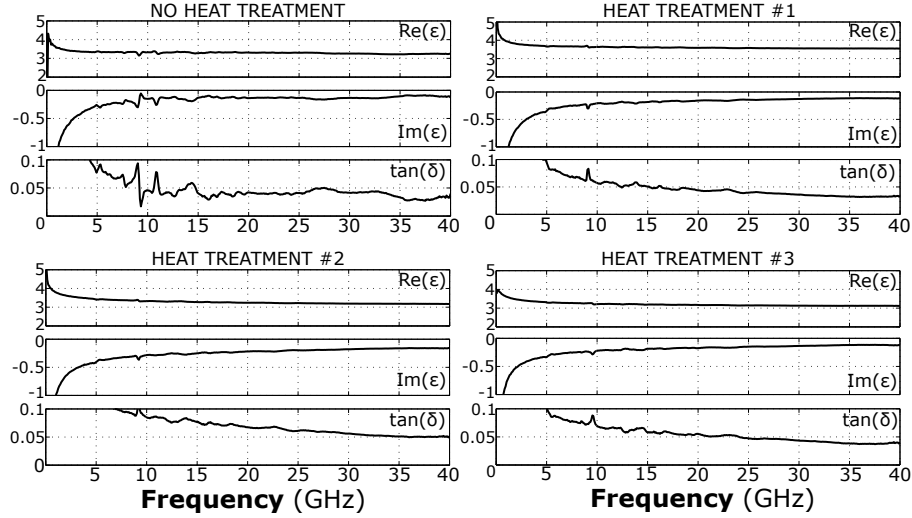


Figure 2.20 – Graphs of the dielectric constants ($\text{Re}(\varepsilon_r)$), the imaginary parts of the permittivity ($\text{Im}(\varepsilon_r)$) and the loss tangent factors ($\tan \delta$) for each kind of *Synkera* AAO membranes (No heat treatment, Heat treatments #1, #2, #3).

2.2.4 Ohmic losses in transmission lines

Another contribution for the insertion losses is the losses that appear in transmission lines. It depends on the skin depth effect resulting on a non-homogeneous distribution of the *ac* current in the conductor. The current runs near the surface of the material, which has a calculated

thickness using the formula [86]

$$\delta_a = \sqrt{\frac{2\rho}{\omega\mu}}, \quad (2.15)$$

where ρ is the resistivity of the conductor, ω the angular frequency of the *ac* current and μ the magnetic permeability of the material. For Au and Cu materials which are used in the transmission lines, δ_a is equivalent respectively to $0.79 \mu\text{m}$ and $0.65 \mu\text{m}$ at 10 GHz, if we increase the frequency up to 40 GHz, these values drop to $0.395 \mu\text{m}$ and $0.325 \mu\text{m}$, respectively. Considering the formula of the resistance of the transmission line $R = \rho l / (w * t)$, where l , w and t are the length, the width and the thickness of the line respectively, t is equivalent to the effective thickness t_{eff} with

$$\begin{aligned} t_{eff} &= t & \text{if } t < 2 * \delta_a, \\ t_{eff} &= 2 * \delta_a & \text{if } t > 2 * \delta_a. \end{aligned} \quad (2.16)$$

We are used to deposit $1 \mu\text{m}$ -thick metallic layers for the transmission line and ground layer (cathode). It means that, if conductive losses appear when the thickness of conductor is less than $2 * \delta_a$, upper than 15 GHz, there is no more conductive losses because $\delta_a(15 \text{ GHz}) = 0.5 \mu\text{m}$ for the Cu layer. The idea is also to investigate the quality of the transmission lines in relation to the high roughness of AAO membranes.

We characterized several stripline thicknesses from $h_{LT} = 0.5 \mu\text{m}$ to $4 \mu\text{m}$. These striplines have been deposited on the same cut NiFe nanowire array with similar lengths, nanowires diameter $\varphi = 35 \text{ nm}$, membrane height $h_p = 50 \mu\text{m}$, porosity $P = 12 \%$ and estimated relative nanowires length $h_w = 0.5$. Transmission results are shown in dB/cm in Fig. 2.21 for three different stripline thicknesses ($h_{LT} = 0.5 \mu\text{m}$, $1 \mu\text{m}$ and $2 \mu\text{m}$). Thickness of the ground layer is $h_G = 1 \mu\text{m}$. Measurements are done without any applied magnetic field, after the saturation of the nanowire array. The FMR absorption is visible at 10 GHz. Local minimum and maximum peaks near 17 GHz and 29 GHz are artifacts in the measurement. Dashed lines are guide for the eyes.

We observe an improvement of the transmission when h_{LT} increases. In the low frequency range ($< 5 \text{ GHz}$), we can see that the conductive losses are smaller in the case of thick striplines that corroborates the theory discussed previously. Better transmission is observed in all the frequency range 40 MHz - 40 GHz. More than 5 dB/cm improvement occurs when the stripline thickness increased from $0.5 \mu\text{m}$ to $2 \mu\text{m}$. Less

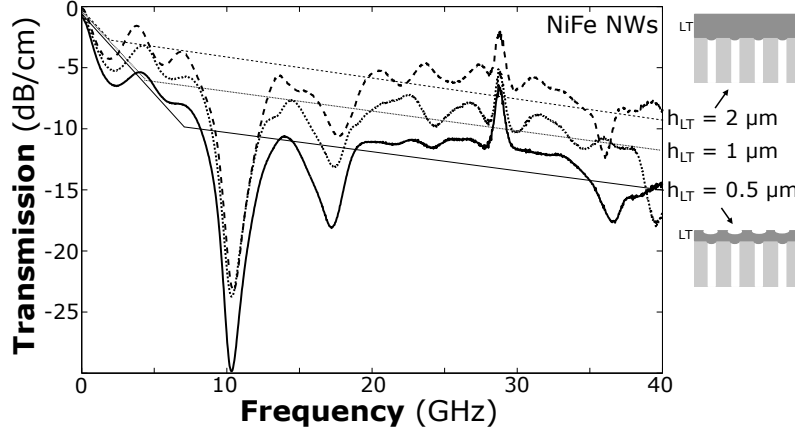


Figure 2.21 – Transmission spectra (in dB/cm) for three stripline thicknesses ($h_{LT} = 0.5 \mu\text{m}$ (solid line), $1 \mu\text{m}$ (dotted line) and $2 \mu\text{m}$ (dashed line)) deposited on the same NiFe nanowire array with nanowires diameter $\varphi = 35 \text{ nm}$, membrane height $h_p = 50 \mu\text{m}$, porosity $P = 12 \%$ and estimated relative nanowires length $h_w = 0.5$. The thickness of ground layer is $h_G = 1 \mu\text{m}$. No external magnetic field is applied and the FMR absorption lies at 10 GHz. Thin lines are guide for the eyes.

roughness is expected in thick striplines, which improves their transmission.

Increasing the stripline thickness above $2 \mu\text{m}$ does not improve the transmission as we can see in Fig. 2.22 where a comparison for two stripline thicknesses are done. The first sample have $h_{LT} = 2 \mu\text{m}$ and the second one $h_{LT} = 4 \mu\text{m}$. We do not observe any improvement in the transmission. In addition, an increase of the ground plane thickness is also compared in Fig. 2.22 with $h_G = 1 \mu\text{m}$ for the first sample and $h_g = 2 \mu\text{m}$ for the second one. No improvement in the transmission is seen. We may conclude that the quality of the layer is already to its maximum with $h_{LT} = 2 \mu\text{m}$. In the case of the ground layer thickness, the sample holder configuration with a metallic plot under the ground avoids the comparison between different thicknesses.

2.2.5 Optimization

The tunability of intrinsic properties is one of the greatest advantages of the magnetic nanowire arrays. The contribution of the magnetic material choice, coupled with specific geometric parameters allows fixing the working frequencies and the magnitude of these reciprocal or

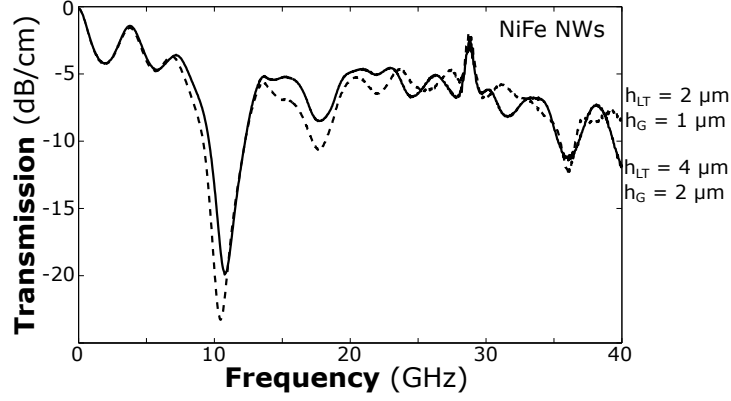


Figure 2.22 – Transmission spectra (in dB/cm) for two stripline and ground layer thicknesses ($h_{LT} = 2 \mu\text{m}$ and $h_G = 1 \mu\text{m}$ (dashed line); $h_{LT} = 4 \mu\text{m}$ and $h_G = 2 \mu\text{m}$ (solid line)) deposited on two NiFe nanowire arrays having similar parameters (nanowires diameter $\varphi = 35 \text{ nm}$, membrane height $h_p = 50 \mu\text{m}$, porosity $P = 12 \%$ and estimated relative nanowires length $h_w = 0.5$).

non-reciprocal effects. Among these intrinsic properties, the theoretical model that permits to control the effective permittivity of nanowired substrates thanks to the nanowires length or to the packing density is of great interest in the conception of isolators, differential phase shifters or circulators. This model will be used to predict numerically the microwave behavior of devices.

Dielectric losses of commercial AAO membranes will be the main issue in the way of an industrial development for the presented microwave devices. As explained in those chapters, high dielectric losses of AAO membranes are the main source of the high insertion losses observed in the transmission. Therefore, an improvement in the fabrication or post-process treatments should be investigated to optimize the dielectric properties of these AAO membranes.

We demonstrated that the quality of the transmission lines is also important for low insertion losses. Thick layers decrease the roughness and improves the flow of microwave signal through the nanowired substrate. $2 \mu\text{m}$ -thick transmission lines on a AAO membrane with 35 nm -pore diameter and porosity $P = 12 \%$ are the best value to achieve the lowest roughness.

Finally, if the performances of microwave devices do not reach the required specifications with the previous solutions, a hybrid topology could be considered to optimize the transmission, like a dielectric mate-

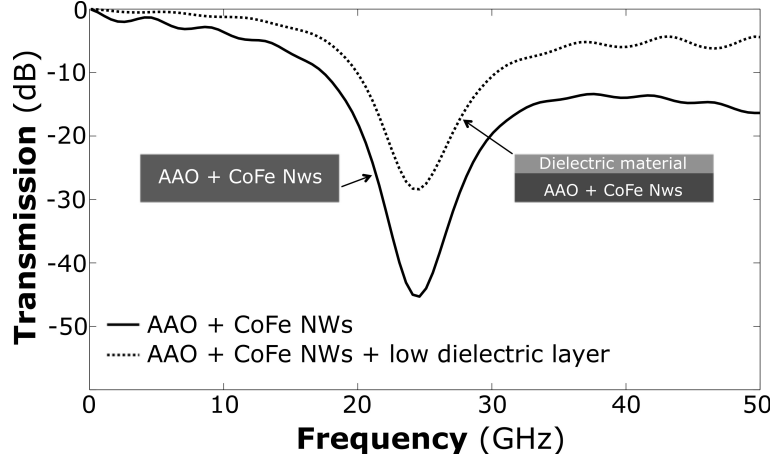


Figure 2.23 – Numerical comparison in the transmission between a hybrid sample (dotted line) composed with 100 μm -thick nanowired AAO membrane and 40 μm -thick low-loss dielectric layer compared with a 140 μm -thick nanowired AAO membrane (solid line). AAO membranes have a porosity $P = 12\%$, dielectric losses $\tan \delta = 0.02$ and is filled with CoFe nanowires with $h_w = 80\ \mu\text{m}$. The dielectric material has $\tan \delta = 10^{-4}$.

rial with low losses ($\tan \delta = 10^{-4}$) deposited on the nanowired substrate. In Fig. 2.23, a hybrid sample composed with 100 μm -thick nanowired AAO membrane and 40 μm -thick low-loss dielectric layer is numerically compared with a 140 μm -thick nanowired AAO membrane. AAO membranes have a porosity $P = 12\%$, dielectric losses $\tan \delta = 0.02$ and is filled with CoFe nanowires with $h_w = 80\ \mu\text{m}$. Great improvements are observed in the transmission of the hybrid sample compared to the single AAO nanowired substrate.

2.3 Magnetic and microwave properties of CoFe and NiFe nanowire arrays

In the purpose to develop nonreciprocal aerospace applications based on our ferromagnetic nanowire arrays, the magnetic remanence of nanowire arrays and temperature fluctuations existing in the space stations or satellites are great issues for many devices. An high remanence ensures nonreciprocal effects, while the temperature stability of microwave and magnetic properties allows working in the space conditions. We wanted to prove the feasibility of aerospace applications with substrates based on

NiFe and CoFe nanowires embedded in nanoporous alumina templates, by demonstrating the high remanence and stability in temperature of their magnetic and microwave properties and also their robustness in temperature. NiFe and CoFe nanowire arrays are grown in the pores of a commercial *Synkera* membrane with various geometric parameters as the pore diameters $\varphi = 18$ nm or 35 nm, the membrane heights $h_p = 50$ μm or 100 μm and porosities $P = 10$ % or 12 %. Their remanences are characterized by alternating gradient measurements and magnetic and microwave properties are characterized through the measurement of a microstrip line at different temperatures in the range -40°C to 80°C , which includes the standard range -15°C to 65°C required for the use of devices in space. In addition, a robustness test in temperature is made in the range -55°C to 125°C .

2.3.1 Hysteresis loops

Hysteresis loops are measured by the alternating gradient method with the saturation field up to 10 kOe. Fig. 2.24 shows the hysteresis loops for different NiFe and CoFe nanowire arrays embedded in AAO membranes with various geometric parameters. The NiFe nanowire array is electroplated into an AAO membrane with pore diameter $\varphi = 35$ nm, membrane height $h_p = 100$ μm and porosity $P = 12$ %. The CoFe nanowire arrays are made into AAO membranes with pore diameters $\varphi = 35$ nm and 18 nm; membrane heights $h_p = 100$ μm , 50 μm , 50 μm and porosities $P = 12$ % and 10 %, respectively.

The magnetic remanence for the NiFe nanowire array is close to 100 %, while the CoFe nanowire arrays of similar pore diameters ($\varphi = 35$ nm) show poor magnetic remanence. A smaller pore diameter ($\varphi = 18$ nm) and lower porosity ($P = 10$ %) CoFe nanowire array allows to improve this remanence close to 100 %. In consequence, CoFe nanowire arrays embedded in AAO membrane with pore diameter $\varphi = 35$ nm should show smaller nonreciprocal effects than the 18 nm-diameter CoFe nanowire arrays.

2.3.2 Temperature dependence of FMR spectra

Measurements are done using a microstrip line method with a VNA after saturating the nanowire arrays with a parallel magnetic field to the wire axis up to 10 kOe. For the NiFe and CoFe nanowire arrays, the FMR absorptions at room temperature have been found around 11 GHz and 24 GHz, respectively. Fig. 2.25(a) shows frequency spectra of NiFe nanowire array at different temperatures, without any applied

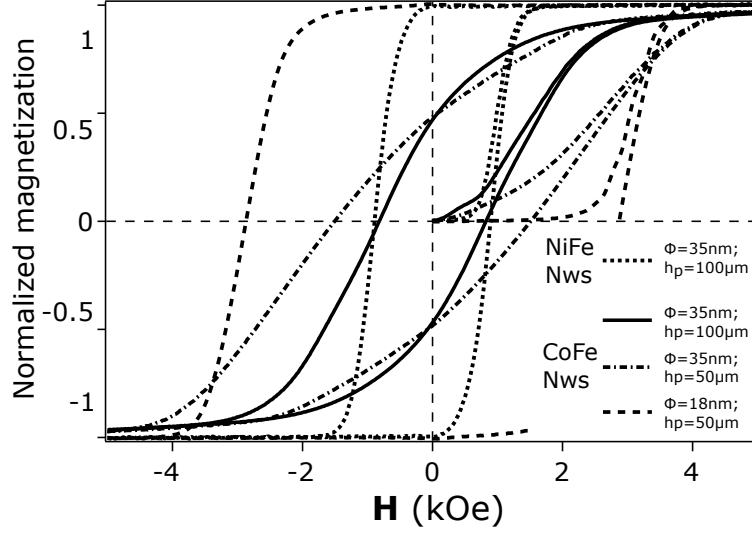


Figure 2.24 – Hysteresis loops for NiFe and CoFe nanowire arrays for different pore diameters or membrane heights. Relative nanowire lengths are in the range [50-70] % for all the samples.

magnetic field. We clearly see the FMR peak close to 11 GHz. The FMR frequencies are shifted in temperature, increasing under cooling while decreasing under heating. This shift is about 800 MHz over a 120°C span.

In Fig. 2.25(b), the CoFe FMR absorption occurs at 24 GHz and the shift in FMR frequencies reaches 1 GHz for 120°C span. The sensibility of the FMR absorption is the ratio between the shift of FMR peak and the FMR frequency at room temperature and is equivalent to 7% for the NiFe nanowire array and 3.5% for the CoFe nanowire array. The dependence of FMR peak is quite linear in temperature and can be explained by the variation of the saturation magnetization of the NiFe and CoFe alloys in temperature. Indeed, taking into account the energetic consideration, the magnetoelastic and the magnetocrystallographic contributions are negligible in comparison with the magnetostatic energy for NiFe and CoFe nanowire arrays. The variation of the saturation magnetization affects the magnetostatic energy $E_{ms} = \pi M_s^2 (1 - 3P)$.

Fig. 2.26 shows the normalization of the saturation magnetization calculated thanks to the data of Fig. 2.25(a), knowing that $M_s = 850 \text{ emu}\cdot\text{cm}^{-3}$ at room temperature (20 °C). These results are compared with a standard measurement of the saturation of magnetization in temperature [95]. For both measured and theoretical curves, a roughly normaliza-

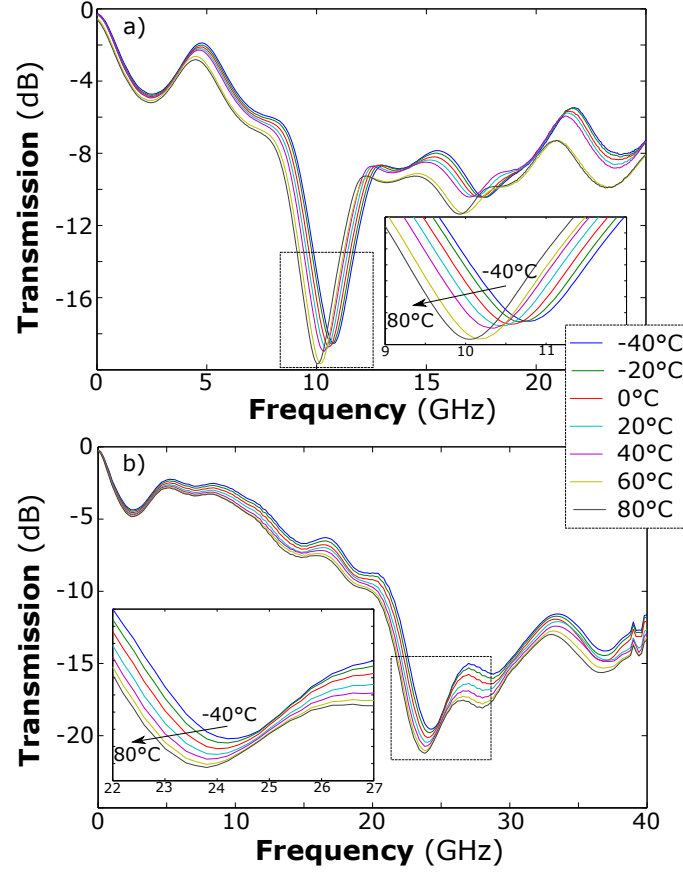


Figure 2.25 – Frequencies spectra measured using a transmission line method of saturated (a) NiFe and (b) CoFe nanowire arrays at different temperatures in the range -40°C to $+80^{\circ}\text{C}$. The FMR absorption lies close to (a) 11 GHz and (b) 27 GHz.

tion is done compared to the highest measured saturation magnetization and not compared to the 0 K. The normalization of the temperature (x -axis) is done compared to the Curie temperature of the NiFe alloys $T_c = 850$ K. The two results show good agreements, which allows to confirm the hypothesis that the shift of FMR peaks comes mainly from the variation of saturation magnetization with temperature.

The robustness test is done in the temperature range -55°C to 125°C without test fixture, in order to preserve it from high temperatures. Measurements at 20°C before and after the temperature cycle (not shown here) report a stability in the microwave and magnetic properties of the NiFe and CoFe nanowire arrays.

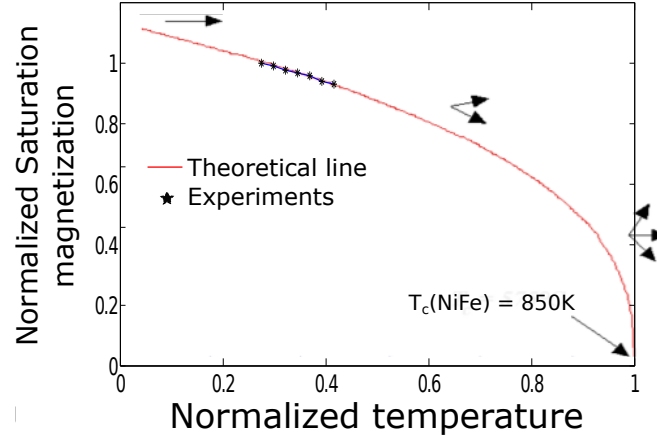


Figure 2.26 – Saturation of magnetization obtained from the measured frequency spectra (Fig. 2.25(a)), normalized to the highest value and to the Curie temperature T_c of NiFe alloy, compared to a normal curve of the saturation of magnetization.

In conclusion, this temperature characterization demonstrated that the NiFe or CoFe nanowire arrays have a good stability of their magnetic and microwave properties. On a 120°C range, the sensitivity of the FMR frequency is only 7% and 3.5% for NiFe and CoFe nanowire arrays, respectively, due to their high Curie temperature. Coupled with the high FMR frequencies of this kind of nanowired substrates, this high Curie temperature allows outperforming the properties of the ferrite materials.

Chapter 3

Nonreciprocal devices based on microstrip topology

3.1 Introduction

Current passive nonreciprocal microwave devices based on ferrite materials [6, 8, 7] have received great attention in the last decades and are nowadays employed in a wide spectrum of applications. However, these devices present limitations since their frequency of operation is usually limited to the X-band due to the low saturation magnetization value of ferrites and they require to be biased by an external magnetic field for their operation which represents a limitation of size reduction. Self-biased hexaferrites may be used for nonreciprocal millimeter-wave devices [8, 13]. They usually require hybrid topology, for example, the hexaferrite disc is mechanically inserted in a circular cavity drilled in a low-loss insulating substrate supporting *rf* access lines, requiring bonding wires to make the contact between hexaferrite cavity and planar accesses. Our nanocomposite based on arrays of magnetic nanowires embedded into a nanoporous membrane presents several advantages: the high length to diameter aspect ratio of low diameter nanowires makes them self biased magnetically along their axis due to the shape anisotropy, and their frequency of operation lies above the X-band, up to 30 GHz in CoFe alloyed nanowire arrays. Nonreciprocal microwave devices based on magnetic nanowired substrate have been the subject of recent works and include circulators [36, 54, 37, 96], isolators [45, 97] and differential phase shifters [40, 42]. In microstrip line topologies lying on a ferromagnetic or ferrite material the microwave electric and magnetic field patterns are non uniform by the virtue of the edge-guided mode since they are preferably concentrated near one long edge of the

microstrip due to the coupling between the microwave signal traversing the microstrip and the gyromagnetic properties of the magnetized magnetic material [98]. The principle combined with an asymmetrical loading of the microstrip results in a nonreciprocal behavior that has recently been applied to obtain differential phase shift performances in a magnetic nanowired substrate [40, 42].

In this chapter we propose a nonreciprocal microstrip line based on a magnetic nanowired substrate, showing a large differential phase shift observed in absence of *dc* magnetic field, and induced by a ferromagnetic resonance transverse field displacement principle. The proposed device consists in a microstrip line geometry on a single planar substrate filled with nanowires of different magnetic alloys, namely NiFe and CoFe. The nonreciprocal behavior of the present device is achieved by controlling the phase velocity of the microwave signal guided on the microstrip line via a variation of the magnetic nanowired substrate permeability. The simulated absorption and differential phase shift are in excellent agreement with the experimental results and the proposed model allows predicting further tunable capabilities of the nonreciprocal microstrip line presented in this chapter.

3.2 Device topology and fabrication

The filling of the membranes with the nanowires is carried out in two steps with the purpose of obtaining two adjacent rectangular zones where CoFe and NiFe nanowires are grown. In step one, electrodeposition of CoFe nanowires takes place in a limited area with width greater than 4 mm and length $L_w = 8$ mm and the NiFe nanowires are subsequently electrodeposited in an area with width greater than 0.5 mm and length $L_w = 8$ mm just besides the one for the CoFe nanowires. For the growth of nanowires, rubber masks are used in order to accurately define each area and to prevent growth of NiFe nanowires over the previously deposited CoFe nanowires. The nanowires height is determined from the time necessary to completely fill the porous membrane. Finally, a 4.5 mm wide and 12 mm long microstrip line is evaporated onto the side of the membrane opposite to the one with the previously evaporated cathode as seen in the nonreciprocal microstrip line photograph of Fig. 3.1. The microstrip line is such that its long dimension is placed parallel to the junction of both zones of nanowires and covers a width $w_c = 4$ mm of the CoFe area and $w_n = 0.5$ mm of the NiFe area as seen schematically in the corresponding 2D and 3D views of the nonreciprocal microstrip line shown in Fig. 3.1 (b)-(c). The taper sections are added in order

to facilitate the contact with the connectors of the test fixture used for the measurement and improve the matching of the wide nonreciprocal microstrip line section to the $50\ \Omega$ reference impedance of the measuring equipment. A large width of the nonreciprocal microstrip line is needed over the nanowire area to induce the field displacement mechanism that will be detailed in the next section. The total length of the microstrip device is $L = L_w + 2L_T = 12\text{ mm}$ with the addition of the taper length $L_T = 2\text{ mm}$.

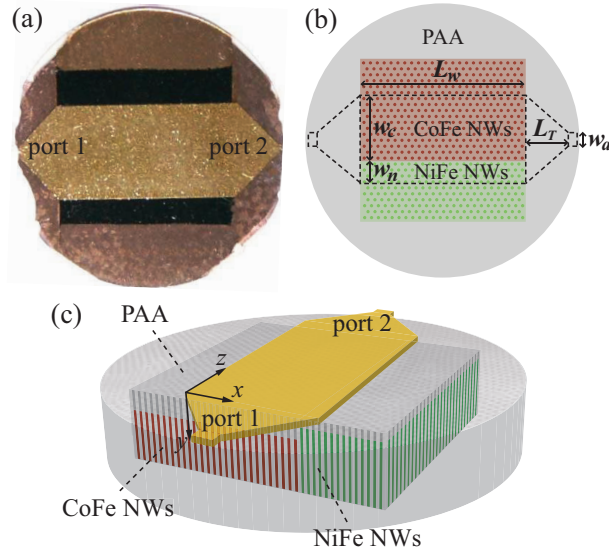


Figure 3.1 – (a) Photograph of the top view of the fabricated nonreciprocal microstrip line. (b) Schematic 2D view of the nonreciprocal microstrip line showing the spatial arrangement of the areas with nanowires and the microstrip line (dotted lines) which is divided in two zones with widths $w_c = 4\text{ mm}$ and $w_n = 0.5\text{ mm}$ and length $L_w = 8\text{ mm}$ over the zones with CoFe and NiFe grown at heights 0.68 and 0.84 ratio of the membrane thickness, respectively. The width of the microstrip line is $w = w_c + w_n = 4.5\text{ mm}$ and the tapes to a width $w_a = 0.2\text{ mm}$ at their two ports and its total length is $L = L_w + 2L_T = 12\text{ mm}$ where $L_T = 2\text{ mm}$ is the taper length. (c) Corresponding 3D schematic view.

All the process of fabrication can be found in chapter 2.1, i.e. the electrolytic solutions parameters listed in table 2.1. The list of samples used in this chapter and its geometric parameters, as well as the fabrication technique for the transmission line, can be found in table 2.2.

3.3 Theoretical predictions

3.3.1 Field-displacement mechanism

The nonreciprocal operation of the device of Fig. 3.1 is based on a transverse field displacement along the microstrip line, which is caused by the ferromagnetic materials beneath the microstrip. The idea was to form areas below the transmission line with different properties, which would achieve the nonreciprocal effect. Contrary to previously reported nonreciprocal microstrip line, where the nonreciprocal behavior depends on an asymmetrical filling with nanowires of the same material [40, 42], the principle is based on the difference of magnetic properties in each area. Therefore the present device is nonreciprocal even if the magnetic nanowired substrate is symmetrically filled with nanowires. The principle of operation of the former depends on the strong variation of height profile of the nanowire array, which can be stairway-like [40] or gradient-like [42], and then on the resulting asymmetry of the substrate permittivity across the microstrip line. In the latter, the principle of operation arises from the asymmetry on the permeability tensor $\bar{\mu}_i$ in each zone, with $i = c$ for CoFe nanowires or $i = n$ for NiFe nanowires. The *rf* signal in the forward direction will have its main energy density located in the area of one kind of nanowires, when in the reverse direction, the energy density will be mostly condensed in the area with the other kind of nanowires. The nonreciprocal operation will result at the FMR frequency of the later area where the *rf* signal will be absorbed in the reverse direction while it will be transmitted in the forward direction. In the presented device, both permittivity and permeability values vary in each zone. Variations in the permeability affect the amplitude of the *rf* signal while the permittivity is varied in order to control the phase velocity of the microwave signal passing through the microstrip line. As a consequence of the presence of ferromagnetic material, the *y*-component of the electric field E_{yi} and the *x*-component of the magnetic field $H_{xi} = (k_z/\omega\mu_0) E_{yi}$, with $i = c, n$, are nonuniform across the *x* position of the transverse section of the microstrip line, which can be rewritten from Hines formalism [98] as

$$\begin{aligned} E_{yc} &= Ae^{-\alpha_c x}, & \text{for } 0 \leq x \leq w_c, \\ E_{yn} &= Ae^{-\alpha_c w_c} e^{-\alpha_n (x-w_c)}, & \text{for } w_c \leq x \leq w_n. \end{aligned} \quad (3.1)$$

In eq. (3.1), $\alpha_i = j\kappa_i k_z / \mu_i$, where k_z is the propagation constant and $j\kappa_i$ and μ_i are the off-diagonal and diagonal components of the permeability tensors, respectively, for zones with CoFe ($i = c$) and NiFe

($i = n$) nanowires and are given by [54]

$$\begin{aligned}\mu_i &= 1 + 2\pi M_{si}\gamma Ph_i f_i (f_i^2 - f^2)^{-1} (m_i^2 + 1), \\ \kappa_i &= 4\pi M_{si}\gamma Ph_i f (f_i^2 - f^2)^{-1} m_i.\end{aligned}\tag{3.2}$$

In the previous equation $f_i = \gamma (H_{dc} + 2\pi M_{si} (1 - 3P)) + j\alpha_{li}f$ is the FMR frequency, $m_i = M_i/M_{si}$ is the normalized magnetization along the y -axis of the nanowires, $\alpha_{li} = 1/(2\pi f\tau_i)$ is the damping factor, H_{dc} is the static field applied parallel to the y -axis of the nanowires which in our case is set to zero, f is the microwave operation frequency, M_{si} is the saturation magnetization, $\gamma = 3 \text{ GHz}\cdot\text{kOe}^{-1}$ is the gyromagnetic ratio, while P is the volumetric fraction of nanowires in the porous template. It is worth mentioning that the difference between the permeability tensors for the CoFe and NiFe nanowired zones arises from the differences between f_c and f_n and, more precisely, from M_{si} and τ_i , which have values $M_{sc} = 1850 \text{ emu}\cdot\text{cm}^{-3}$, $M_{sn} = 825 \text{ emu}\cdot\text{cm}^{-3}$, $\tau_c = 6.3 \cdot 10^{-11}$, and $\tau_n = 1.46 \cdot 10^{-10}$. Equation (3.1) enables us to predict that the decay of the field along the positive x -direction is different in the two zones because of the different permeabilities of the material, but also that this decay is reversed, i.e., occurring along the negative x -direction or, equivalently, causing amplification in the positive x -direction, when the direction of either propagation (fixing the sign of k_z , hence of α_i) or magnetization (fixing the sign of m_i) are inverted. The FMR mechanism implies that the sign of components κ_i is different below and above FMR frequency, meaning that the decay of the field is reversed from below-resonance to above-resonance frequency range. These predictions are illustrated and confirmed at Fig. 3.2, showing the field patterns simulated for the device presented in this section, using eq. (3.1).

The nonreciprocal behavior is achieved by combining this field displacement mechanism with the change of permeability across the transverse section induced by the change of ferromagnetic material : as the field concentration in each zone depends on the propagation (or magnetization) direction, so will the effective permeability experienced by the microwave signal guided also depend on the microstrip line. Hence, its quasi-TEM propagation constant and characteristic impedance will depend on the sense of propagation (or magnetization), making the device nonreciprocal.

3.3.2 Quasi-TEM nonreciprocal transmission-line formalism

Using the effective medium approach, an analytical homogenization formula is proposed for the effective permeability experienced by the

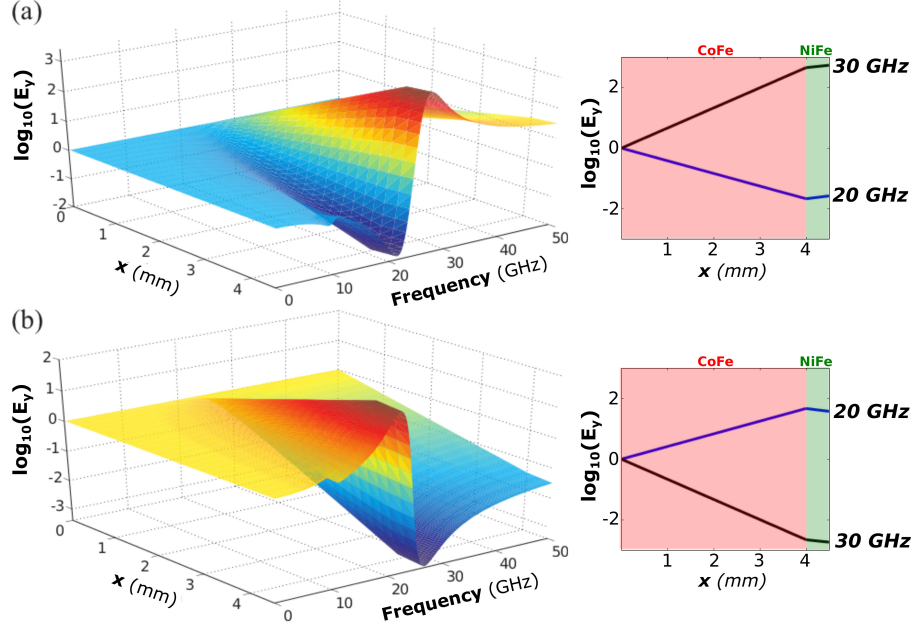


Figure 3.2 – 3-D view (left side) of the calculated electric field pattern E_y as a function of the x -position and the excitation frequency f at zero external bias field for (a) positive and (b) negative direction of propagation (or magnetization). 2-D view (right side) of the calculated E_y as a function of the x -position for certain frequencies, below (20 GHz) and above (30 GHz) CoFe FMR frequency for both directions of propagation. The background colors represent the CoFe (red) and NiFe (green) zones. Log magnitude of field is plotted for sake of clarity, after normalization for each frequency to its value at the side edge of CoFe nanowire area ($x = 0$). As magnetic field H_x is proportional to E_y by the factor $k_z/\omega\mu_0$, its pattern looks identical to the E-field pattern after normalization.

signal for each direction of propagation (or magnetization), along positive (+) or negative (-) z (or y) axis:

$$\frac{1}{\mu_{eff}^{\pm}} = \frac{\int_0^{w_c} \frac{\mu_c}{\mu_c^2 - \kappa_c^2} H_{x_c}^{\pm}(x) dx + \int_{w_c}^{w_n} \frac{\mu_n}{\mu_n^2 - \kappa_n^2} H_{x_n}^{\pm}(x) dx}{\int_0^{w_c} H_{x_c}^{\pm}(x) dx + \int_{w_c}^{w_n} H_{x_n}^{\pm}(x) dx}, \quad (3.3)$$

where the expression $(\mu_i^2 - \kappa_i^2)/\mu_i$ is the effective permeabilities associated with the quasi-TEM propagation in each ferromagnetic zone [99]. The use of inverted quantities is required in eq. (3.3) because inductive contributions under the microstrip have to be considered in parallel and not in series [100]. Superscripts $\pm$ in the field variables indicates that eqs. (3.1) are used either with positive/negative values of propagation

coefficient ($\Im(k_z) = \pm \|\Im(k_z)\|$) for positive value of $\Im(k_z)$. From expression (3.3), nonreciprocal quasi-TEM complex propagation constants depending on direction of propagation (or magnetization), and associated characteristic impedances can be calculated as

$$k_{\pm} = j \frac{\omega}{c_0} \sqrt{\mu_{eff}^{\pm} \epsilon_{eff}}, \quad (3.4)$$

$$Z_{c\pm} = 377 \frac{h}{w} \sqrt{\frac{\mu_{eff}^{\pm}}{\epsilon_{eff}}}, \quad (3.5)$$

where ϵ_{eff} is the equivalent effective permittivity of the nanowired substrate, function of the filling factor [35], w is the width of the microstrip over the nanowire area, and h is the thickness of the alumina membrane.

3.4 Experimental validation

The transmission coefficient $T_{\pm} = e^{k_{\pm}L}$ in the forward (+) and backward (−) propagation directions has been simulated using the formalism summarized in the previous section at zero applied dc bias magnetic field ($H_{dc} = 0$). The S –matrix of the nonreciprocal microstrip line section lying on the nanowired part of the substrate was calculated from $T_{\pm}$ and $Z_{c\pm}$ using the formalism proposed in [101] for nonreciprocal transmission lines. It was then transposed to the S –matrix referenced to the 50 Ω of the measuring equipment using classical S – to chain-matrix conversion formulas, and making a cascaded product of chain matrices of input taper, nonreciprocal microstrip line nanowired section, and the output taper, respectively. Microwave measurements are carried out in two-port configuration using a vector network analyzer (VNA) (Anritsu 37297C). The ($\pm$) propagation directions in experiments correspond to propagation from port 1 to port 2 and from port 2 to port 1, respectively, as shown in Fig. 3.1(c). Fig 3.3(a) shows, in both directions of propagation, the magnitude $S_{\pm}/L$ of the transmission S –parameters normalized per unit length, expressed in dB·cm^{−1}. A significant isolation between forward and reverse S –parameters is observed around the resonance frequency for CoFe at $f_c = 22.32$ GHz, in both experimental and simulated curves. In simulations, the loss tangent factor is set to $\tan \delta = 0.067$, corresponding to the commercial Synkera substrate, which explains the high insertion losses over the whole frequency range (see section 2.2). Such a high value for dielectric losses in alumina is explained by the imperfect purity of the commercial membrane resulting from the anodization fabrication procedure, which might also allow the presence of residual bound water molecules in its nanoporous structure

[93, 94]. A more significant FMR absorption is present in both propagation directions around the f_c frequency and not at $f_n = 9.95$ GHz, which may be ascribed to the fact that $M_{sc} > M_{sn}$ and then the absorption due to the NiFe nanowires is much lower than that for the CoFe nanowires. Furthermore, the isolation of about 18 dB observed around this frequency between forward and reverse transmission clearly indicates a nonreciprocal behavior of the device. It is worth mentioning that resonant transmission peaks observed below 15 GHz are due to imperfect matching offered by tapered edges of the microstrip line at low frequency: because of their small length ($L_T = 2\text{mm}$) with respect to corresponding wavelengths, quarter-wavelength matching to $50\text{-}\Omega$ impedance is not ensured below 15 GHz.

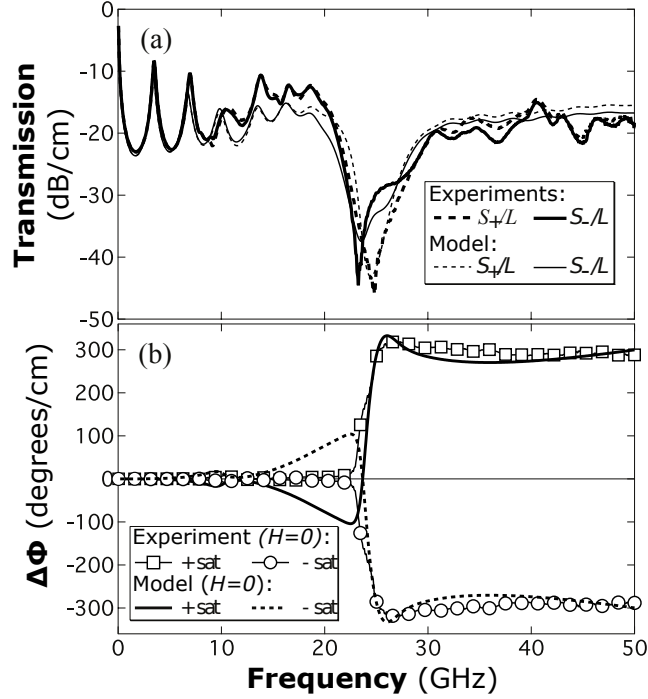


Figure 3.3 – (a) Measured (tick lines) and calculated (thin lines) S_{+}/L (dashed lines) and S_{-}/L (continuous lines) for the nonreciprocal microstrip line of the Fig. 3.1(b) at zero dc biasing magnetic field. (b) Measured (symbols) and calculated (lines) $\Delta\phi/L$ at zero field for the nonreciprocal microstrip line of (a).

Interestingly, the measured differential phase shift per unit length $\Delta\phi/L = (\varphi_{+} - \varphi_{-})/L$ of the nonreciprocal microstrip line of Fig. 3.3(b) (squares), where $\varphi_{\pm}$ is the phase of the $S_{\pm}$ parameter, is negligible

for $f < f_c$ but suddenly increases for $f > f_c$ up to the value 300 degrees·cm⁻¹. Furthermore this value is higher than those reported for devices based on ferrites in the range 38-130 degrees·cm⁻¹ at X -band and 300 K [102, 103, 104, 105]. It is worth mentioning that the high $\Delta\varphi/L$ value obtaining without the application of a dc magnetic field in our device is suitable for the realization of a gyrator element ($\Delta\varphi = 180$ degrees) operating in the frequency range 30-50 GHz, which can be obtained with a shorter microstrip line having a length of 0.72 cm. The $\Delta\varphi/L$ calculated from the $S_{\pm}$ -parameters (continuous line) predicts negative values close below f_c that are not observed experimentally, however, it agrees well with the measured one above this frequency value. As explained before, either if the sign of the propagation constant k_z is reversed by inverting the propagation direction, or the sign of the off-diagonal term of the permeability tensor $j\kappa_i$ is changed by reversing the magnetization direction, an inversion of the field patterns occurs. Particularly, inverting the sign of $j\kappa_i$ by virtue of a change of sign in m_i (see eq. (3.2)) leads to an inversion of $\Delta\varphi/L$ as seen by the dashed line in Fig. 3.3(b). Experimentally, $\Delta\varphi/L$ (circles) is inverted by magnetizing the nonreciprocal microstrip line via the application of a saturating dc field in the opposite direction which is subsequently relaxed to zero. As seen in Fig. 3.3(b), the most striking feature is that $\Delta\varphi/L$ remains practically constant above f_c where insertion losses are reduced with respect to those at the FMR absorption for CoFe nanowires. This means that, in that frequency range, the present device has improved performances as a nonreciprocal phase shifter. As seen in Fig. 3.4(a), the figure of merit (FOM) = $2\Delta\varphi / (S_+ + S_-)$ reaches values of about ± 15 degrees·dB⁻¹ in the frequency range 30 - 50 GHz, and both the experimental (continuous line) and calculated (dotted line) values are in good agreement.

3.5 Parametric study and optimization of the present device

The good agreement between experimental and calculated insertion losses and differential phase shift observed in Fig. 3.3 suggests that the proposed model allows to predict with good accuracy the microwave properties of our nonreciprocal microstrip line. Simulations in Figs. 3.3 and 3.4(a) take into account the ohmic losses of microstrip and ground metallization as well as the conductivity of the nanowires. An UCL research group have however demonstrated previously in [35] that the loss tangent factor of nanowired substrate does not increase with the presence of conductive metallic nanowires far away from the ferromagnetic reso-

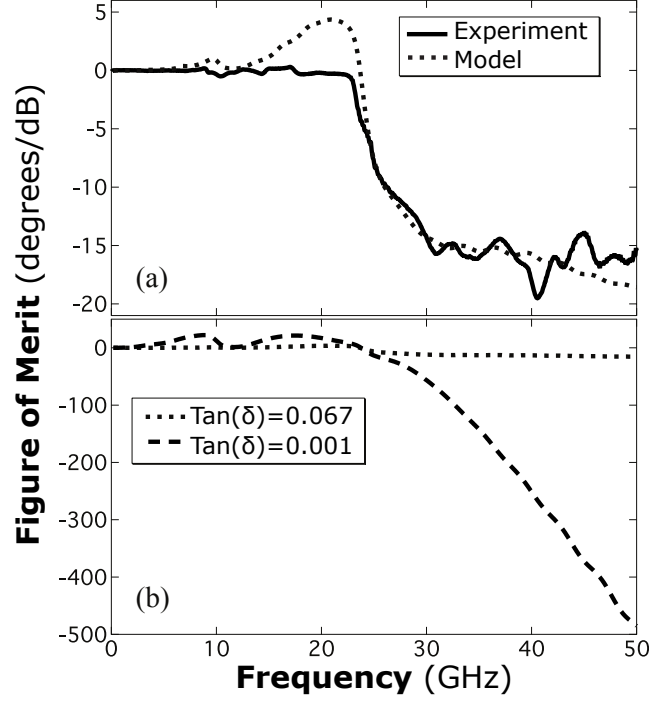


Figure 3.4 – (a) Measured (continuous line) and calculated (dotted line) FOM at zero field after saturating the nonreciprocal microstrip line of Fig. 3.1(a) in a positive magnetic field. (b) Calculated FOM for $\tan \delta = 0.067$ (dotted line, same as dotted line in Fig. 3.4(a) for actual device) and for $\tan \delta = 0.001$ with perfect matching of nonreciprocal microstrip line (dashed line).

nance. Improving the matching (i.e., optimizing the geometry of tapers to ensure a perfect matching to 50Ω) will also reduce insertion loss. High dielectric loss reported in the literature for porous alumina is thus the remaining issue to solve, for example, by increasing alumina purity and performing some annealing to avoid residual moisture (see section 2.2). FOM performances can be further improved by reducing insertion losses on $S_{\pm}$ that are mainly due to the dielectric losses of the porous anodic alumina membrane, with a residual part induced by mismatch. Indeed, higher FOM of up to $480 \text{ degrees}\cdot\text{cm}^{-1}$ can be obtained using a high-purity porous anodic alumina membrane with $\tan \delta = 0.001$, together with a perfect matching of the nonreciprocal microstrip line section to 50Ω (Fig. 3.4(b)). Therefore, improved performances are expected for lower $\tan \delta$ values in the frequency range above the main FMR absorption, which in this case corresponds to the one for the CoFe nanowires.

3.5.1 Width of nanowire areas

As an example, our model also shows that these properties can be tuned just by moving the position of the microstrip line in the x -direction perpendicular to the propagation direction or equivalently by changing the widths of the NiFe and CoFe nanowired zones beneath the microstrip. The tunable feature of the nonreciprocal microstrip line of Fig. 3.1 is observed in Fig. 3.5(a) from the variation of $\Delta\varphi/L$ with the width w_n (numbers at the right vertical axis) of the NiFe nanowired zone via the relation $w_n = w - w_c$ with $w = 4.5$ mm.

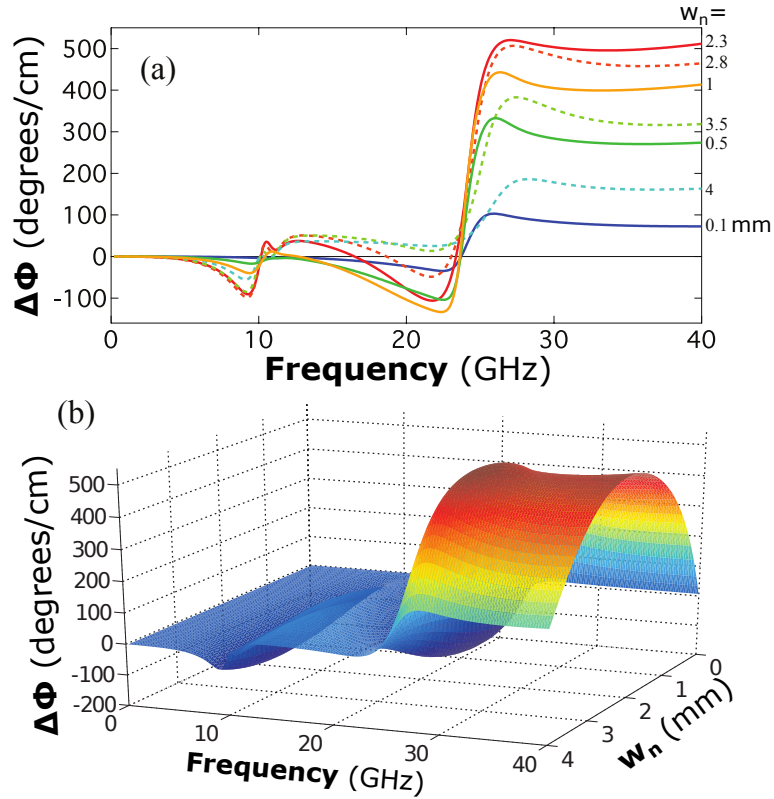


Figure 3.5 – Calculated $\Delta\varphi/L$ as a function of frequency and width w_n of the NiFe nanowired zone such that $w = w_n + w_c = 4.5$ mm, with w_c the width of the CoFe nanowired zone. (a) 2-D view. (b) 3-D view. Numbers in (a) correspond to values of w_n in millimeters.

As observed in this figure, $\Delta\varphi/L$ increases in the frequency range $f > f_c$ as w_n increases below 2.3 mm (continuous lines) and decreases above this value (dashed lines). The maximum attainable $\Delta\varphi/L$ value is predicted to be about $500 \text{ degrees}\cdot\text{cm}^{-1}$ at $w_n = 2.3$ mm, which means

that a similar density of energy from the rf signal passing through each zone for forward and reverse propagation directions will optimize the differential phase shift. This translation can be reached just by adjusting during the design the position of the microstrip line along the x -axis of the structure instead of adjusting its length L_w . A 3-D view of the variation of $\Delta\varphi/L$ on f and w_n is shown in Fig. 3.5(b), where it is seen that introducing a magnetic material beneath the microstrip line with different magnetization than the one already present leads to significant changes in $\Delta\varphi/L$ for $f > f_c$, where insertion losses are lower. Keeping constant the height of both the NiFe and CoFe nanowires allows fabricating a tunable phase shifter where $\Delta\varphi$ can be varied using the same microstrip line as mentioned above.

3.5.2 Lengths of nanowires

Another parameter easily tunable in the fabrication of the device and having high consequences on the value of the differential phase shift is the relative nanowire length in the two zones. As we know, the nanowire length is the main variable of the calculation of the effective permittivity [35] and it was used in the previous researches on the nonreciprocal microstrip line [40]. For the present device, the relative lengths of the nanowires in both areas are, as mentioned in the Fig. 3.1, 68% and 84% for CoFe and NiFe nanowires, respectively. This difference reinforces the nonreciprocal effect of the device, while two intrinsic properties of the substrate, the effective permeability and the effective permittivity, vary in the two zones. Fig. 3.6 shows the dependence of the $\Delta\varphi/L$ on the two lengths of CoFe and NiFe nanowires at $f = 40$ GHz, while keeping the other parameters constant: $w = w_n + w_c = 4.5$ mm, with $w_n = 0.5$ mm and $w_c = 4$ mm, with positive magnetization and propagation directions.

The highest positive value of differential phase shift is observed for long NiFe nanowires while CoFe nanowires length is keeping in the small range. Large negative values can also appear for long CoFe nanowires while NiFe nanowires have relative length in the range 50 %. $\Delta\varphi/L$ is close to zero (black line) when the two zones have similar nanowire lengths and has an opposite sign while the CoFe nanowires or NiFe nanowires have the longest length. This can be understood seeing the position of each area below the microstrip line, making faster the forward or reverse direction transmission. When the length value of NiFe nanowires are high and the length value of CoFe are low, the differential phase shift can be worn up to $800 \text{ degrees}\cdot\text{cm}^{-1}$.

It is worth to be mentioned that the $\Delta\varphi/L$ performances of our de-

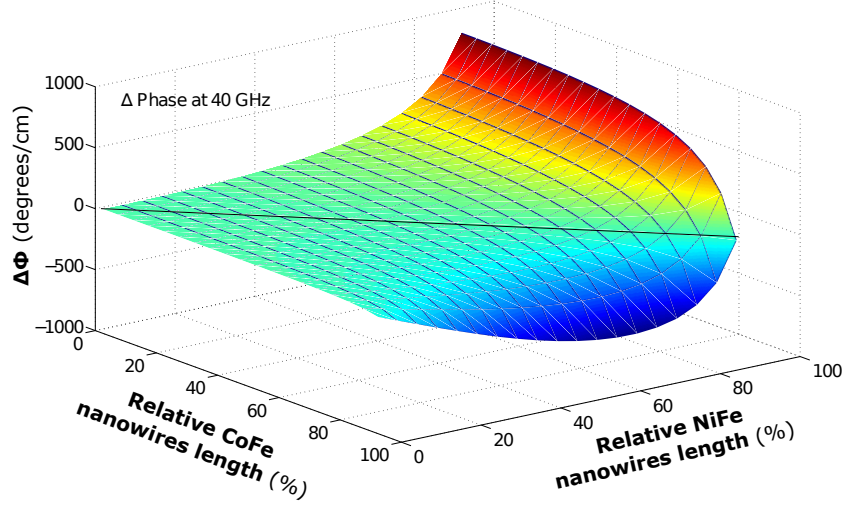


Figure 3.6 – Calculated $\Delta\varphi/L$ as a function of relative nanowires lengths in both areas, when $w_n = 0.5$ mm and $w_c = 4$ mm. The black line shows the value of the $\Delta\varphi/L$ where the both lengths are equivalent.

vice are comparable to those reported for ferrite-superconductor devices [106, 107] for which $\Delta\varphi$ is as high as 1000 degrees in a 2.5 cm-long meander line leading to $\Delta\varphi/L$ as high as 400 degrees·cm⁻¹ at X -band. Even if the FOM of our device is still low, due to the loss tangent factor of the commercial alumina membrane and its not yet optimized matching, our device has the advantage that its frequency of operation lies at Ka -band, suitable for high-frequency microwave applications.

3.6 Microwave isolator and gyrator for space applications

The purpose of this work, linked to the project done in collaboration with *Thalès Alenia Space*, is to investigate the possibility of designing optimized devices for space applications based on a technology built on a nanowire array embedded in nanoporous alumina template. Specifically, two frequency bands have been investigated: Ku band for emission (17.3 - 20.2) GHz and reception (27 - 30) GHz, and Q, V band for emission (37.5 - 42.5) GHz and reception (47.2 - 50.2) GHz. Microwave isolation and gyration (180 degrees-differential phase shift) was searched for each frequency band. We have seen in the previous section that the used theoretical model can predict with good accuracy the microwave

properties of the nonreciprocal microstrip line. Various numerical simulations have been done varying the geometrical parameters as explained in the previous section: the part of each zone below the transmission line, called width ratio, and the relative nanowires lengths in each zone. We also change magnetic materials inside the nanowired substrate: in addition to the CoFe/NiFe nanowired substrate used for the experimental part, we proceeded with simulations using NiFe nanowires coupled with Ni nanowires, with $M_s(\text{Ni}) = 485 \text{ emu}\cdot\text{cm}^{-3}$. We fixed the length of the device to 1 cm and the width to 4.5 mm. Table 3.1 shows all the fixed and variable parameters. For this study, we used the ideal case for the device: a low-loss dielectric nanoporous alumina membrane, with the $\tan \delta = 0.001$ together with a perfect matching of the nonreciprocal microstrip line section to 50Ω .

Fixed parameters
Length of the device : $L = 1 \text{ cm}$
Width of the device : $w = 4.5 \text{ mm}$
Variable parameters
Magnetic materials ($mat1/mat2$):
CoFe / NiFe
NiFe / Ni
Width ratio:
$R = w_1/w = (1 \ 3 \ 5 \ 7 \ 8)/9$
Relative nanowires lengths:
$h_1 = 20\% \text{ to } 90\%$ with gap of 10%
$h_2 = 20\% \text{ to } 90\%$ with gap of 10%

Table 3.1 – Fixed and variable parameters used for the numerical study.

Thanks to a line code in the simulations program, we can select the parameters where the isolation is higher than 15 dB or when the differential phase shift is equivalent to 180 degrees in all the required frequency bands. We present in the Fig. 3.7 the best result for the isolation that only demonstrates in the (27 - 30) GHz frequencies band. The isolation is fixed at the FMR frequency of the CoFe nanowires. The energy of the rf signal in the reverse direction S_- is confined in the CoFe nanowire area, which absorbs the signal in this direction while the signal in the forward direction S_+ is transmitted. The high value of the isolation (more than 60 dB) is due to the large volume of magnetic material in both zones ($h_1 = h_2 = 90\%$), that ensures a strong nonreciprocal effect. This high isolation, obtained with same nanowires length in both zones

prove the high nonreciprocal effect of these devices even if the differential phase shift is close to zero when both nanowires lengths are equivalent in the two zones.

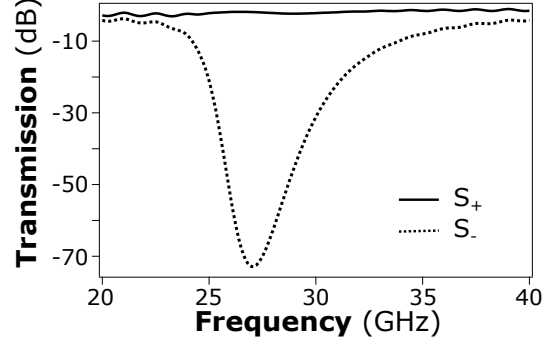


Figure 3.7 – Calculated forward (continuous line) and reverse (dotted line) transmission for a nonreciprocal microstrip line at the 27 - 30 GHz frequency band.

About the differential phase shift, we were able to determine parameters to obtain a gyration in each frequency band (Fig. 3.8). The insertion losses observed in the frequencies range are weak, with values equivalent or lower than 0.5 dB, excepted for the 17.2 - 20.2 GHz frequencies band where the absorption of the NiFe FMR is too close. In Fig. 3.8, the differential phase shift is represented by the long-dashed curves and the forward and reverse transmission by the continuous or dotted curves. Variations of the differential phase shift as a function of the frequency are visible in the Fig. 3.8, which proves the parametric dependence of the value and the behavior of the phase shift. Annotations above each graphs indicate the parameters used for numerical simulations: NiFe/Ni are the magnetic material used for nanowires in each zone of the device, W_{tot} is the width of total transmission line, R is the width ratio for the NiFe width W_1 compared to W_{tot} , h_1 and h_2 are the relative nanowires lengths for zones 1 and 2, respectively.

These numerical results that rely on the experimental confirmation provided in previous section prove the feasibility to design differential gyrators for spatial applications based on unbiased magnetic nanowired substrates. The relatively low insertion losses observed at each frequency band, and in addition the good stability in temperature and the possible use of microelectronic techniques for the fabrication of nanowire arrays permit integrating this kind of device on a circuit. High isolation and relatively low insertion losses observed in the 27 - 30 GHz frequency band for CoFe/NiFe devices give hope to conceive microwave isolators

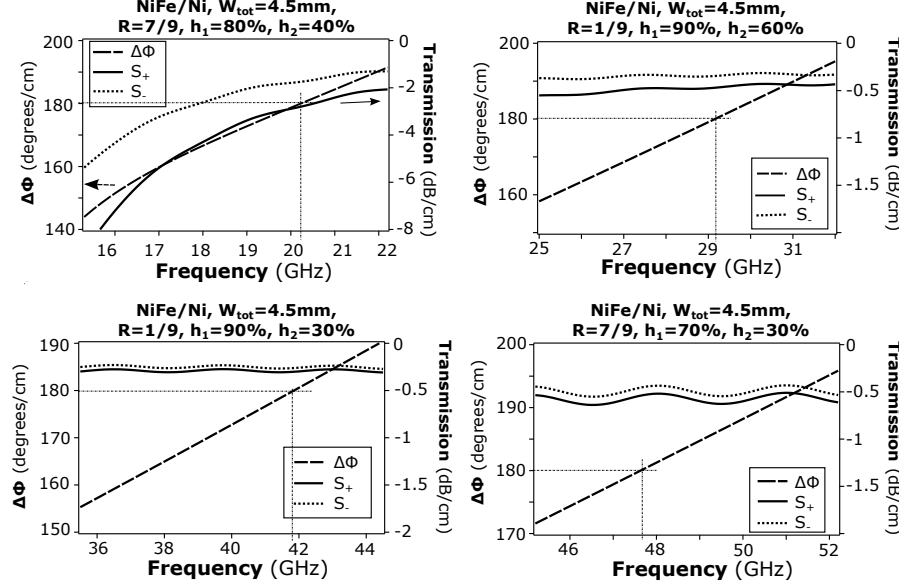


Figure 3.8 – Calculated differential phase shift (long-dashed lines) and transmission for the forward (solid line) and reverse (dotted line) direction in each required frequency band for a nonreciprocal microstrip line. Values above each graph are the parameters used for numerical simulations (see table 3.1).

at specific frequencies close to FMR frequencies of magnetic nanowires.

3.7 Conclusions

Magnetic nanowired substrates have been used for the fabrication of a planar nonreciprocal microstrip device. As a result a nonreciprocal differential phase shift up to $300 \text{ degrees} \cdot \text{cm}^{-1}$ is obtained at zero applied dc magnetic field, making the present device suitable for gyrator applications [108] and electronically phased arrays antennas [102]. The nonreciprocal operation is achieved by loading the device with nanowires of different ferromagnetic materials and different lengths, which allows to control the phase velocity of the microwave signal passing through the device by virtue of the spatial variation of the magnetic substrate permeability. Microelectronic techniques, as e-beam deposition, plasma etching or lithography, should allow machining parallel areas with precise interface. The measured microwave performances of the device have been reproduced with excellent accuracy using a proposed analytical model based on an effective medium theory and useful for the predic-

tion of further tunable capabilities. Optimizations on the width ratio of each zone and on the nanowires lengths provide the understanding of the dependence of differential phase shift on the geometric parameters. A three-areas configuration filled with different magnetic materials was considered to enhance the nonreciprocal behavior, but due to the exponential distribution of the rf signal below the transmission line, which condenses the main part of the energy on the side, the difference of the magnetic properties induces by the area of the middle part is negligible. No improvement of the nonreciprocal effect should be observed considering the three-areas configuration. High figure of merit value is numerically predicted in the case of ideal devices based on low dielectric losses AAO membrane and $50\ \Omega$ -impedance matching. Numerical characterizations of the differential phase shifter allow to transform the device in a microwave isolator working in the Ka -band with required performances, as well to find nanowires nature and geometric parameters to obtain gyration in each frequencies bands required for aerospace applications with industrial performances.

Chapter 4

Nonreciprocal three-ports junction topology

4.1 Introduction

In this chapter, we focus on a three-ports junction topology that can be used in different applications. First, we study the design, the fine-tuning and the optimization of a microwave circulator based on nanowired substrate and working at dedicated frequencies in the X -band (8-12 GHz) for airborne applications. This work is part of a project in collaboration with *Thalès Systèmes Aéroportés*, which is based in Palaiseau, France. The purpose is to conceive and fabricate a microwave circulator working in the X -band with the required standard specifications. This device should have the following performances to answer the commercial standards; isolation higher than 15 dB and insertion loss lower than 2 dB. To achieve this goal, we characterized the dependence of the microwave behavior of the circulator to the nanowires length. In this way, it is possible to control precisely the circulation frequencies, through its influence on the effective permittivity of the substrate. We illustrated this dependence by a successful comparison between measurements and numerical predictions obtained for circulators having various geometrical parameters and ferromagnetic nanowired materials. Then numerical parametric characterizations, as the geometry or the nature of the nanowired substrate or the dimensions of the circulator design, is done to select the parameters that give the best results in order to fabricate a microwave circulator. Finally, a numerical study of an ideal nanowired substrate and circulator design proves the feasibility of fabricating a microwave circulator working in the X -band with the required specifications. Secondly, a special configuration which applies a short-circuit on the

third port, allows to transform this device to a two-port design, which gives the possibility to develop an isolator. A short study done in collaboration with *Thalès Alenia Space*, in Toulouse, France investigated the feasibility to conceive microwave isolators for space applications based on a technology built on a nanowire array embedded in a nanoporous alumina membrane. Parametric simulations result in the finding of all variable values to obtain an isolation in the required frequency ranges. Experimental results bring the confirmation of microwave isolation in most of frequency bands.

4.2 Experimental procedures

The filling of the membranes with nanowires is carried out in one step with the purpose of obtaining a circular zone where CoFe or NiFe nanowires are grown. Laser micromachining system is used to design the circulator transmission line in the Cu/Au layer previously deposited onto the side of the membrane opposite to the one with the evaporated cathode. This method allows a precise definition of the metallic disc pattern of the circulator and its three access lines (Fig. 4.1(a)). The three-ports junction device, shown in the picture of Fig. 4.1(a), is burnt with a 200- μm wide passes of laser micromachining system. The device is in the microstrip configuration since the ground layer is deposited on the other side of the membrane. The remained gold layers, outside of the circulator design, are floating electrodes, therefore no rf field is expected to align with the coplanar configuration. From the SEM image in inset of the Fig. 4.1(b), we can also observe that nanowire arrays form a dense structure, with a uniform filling height of the pore. The variance of the distribution and filling of pores are relevant and sufficiently accurate for microwave predictions. Values of porosity extracted from FMR measurements [23] are also introduced in models used in the next section for these predictions. From the SEM picture, we can see that the cathode acting as ground plane metallization is of uniform thickness, offering good conditions for preventing ohmic losses due to poor surface roughness of the metallization. This three-ports junction topology can be either used as a three-port device or as a two-ports device by applying a short-circuit on one of the port.

The choice of CoFe and NiFe nanowires is the consequence to the characterization in chapter 2.3 where large magnetic remanence and good stability in temperature were observed for both kind of nanowire array. All the process of fabrication can be found in chapter 2.1, i.e. the electrolytic solutions parameters listed in table 2.1. The list of samples used

in this chapter and their geometric parameters, as well as the fabrication technique for the transmission line, can be found in table 2.2.

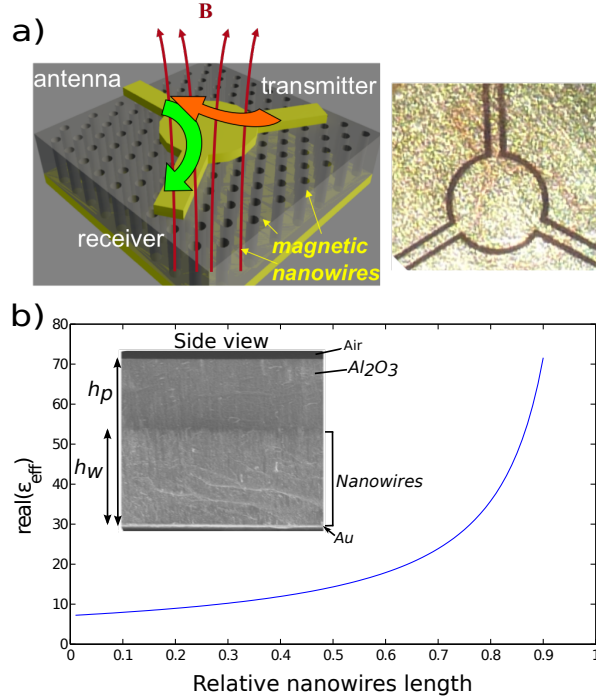


Figure 4.1 – (a) Sketch and picture of microwave circulator based on unbiased ferromagnetic nanowire arrays embedded in a porous anodic alumina membrane. (b) Dependence of the effective real permittivity of nanowired substrate with nanowires length. In inset, a side SEM picture of nanowired substrate with a nanowires length and a membrane height, respectively, h_w and h_p .

4.3 Theoretical model

The model of the microwave circulator is based on the Bosma theory [56] and explained in the case of substrate composed by magnetic nanowires embedded in a dielectric nanoporous membrane by Saib [36, 54]. The model takes into account the planar microstrip geometry, ohmic losses of metallic layers, dielectric losses of the alumina membrane and the impedance matching network connected to the ports of circulator, as well as the intrinsic properties of the nanowired substrate in the remanent state.

4.3.1 Intrinsic properties of nanowired substrate

As explained previously in the section 2.2.2 and reported in [35], the effective permittivity of a nanowired substrate depends on the length and the packing factor of the nanowires. As we used membranes with the same packing factor P for all our measurements, we only consider the change in permittivity due to the nanowires length h_w . In Fig. 4.1(b), we can see the dependence of the real part of effective permittivity due to the nanowires length. As reminder, the effective permittivity of a nanowired substrate is given by the formula:

$$\epsilon_{eff} = h_p \left(\frac{h_w}{\epsilon_w} + \frac{h_a}{\epsilon_p} \right)^{-1}, \quad (4.1)$$

where h_p is the height of the membrane, h_w the length of the nanowires, h_a the gap between the top of nanowires and the surface membrane, ϵ_p and ϵ_w the permittivity for the air gap and the nanowired part defined as:

$$\epsilon_p = P + (1 - P) \epsilon_b (1 - j \tan \delta), \quad (4.2)$$

$$\epsilon_w = P \left(1 - j \frac{\sigma_w}{\omega \epsilon_0} \right) + (1 - P) \epsilon_b (1 - j \tan \delta), \quad (4.3)$$

where j is the imaginary unit, σ_w the conductivity of the wire, $\tan \delta$ the dielectric loss tangent factor of porous alumina and ϵ_b its dielectric constant.

For the magnetic properties of the substrate, as mentioned in the section 1.2.3, the permeability tensor is given by:

$$\mu_{eff} = \begin{pmatrix} \mu & -j\kappa & 0 \\ j\kappa & \mu & 0 \\ 0 & 0 & 1 \end{pmatrix}, \quad (4.4)$$

with

$$\mu = 1 + 2\pi M_s \gamma P h_w f_r (f_r^2 - f^2)^{-1} (m^2 + 1), \quad (4.5)$$

$$\kappa = 4\pi M_s \gamma P h_w f (f_r^2 - f^2)^{-1} m, \quad (4.6)$$

where M_s is the saturation magnetization, P the packing factor, h_w the length of nanowires, f_r the ferromagnetic resonance frequency and $m = M/M_s$ the normalized magnetization.

4.3.2 Solution of the wave equations

As an approximation of a thin layer, the circulator has a cylindrical symmetry, with no z -dependence to the magnetic or electric fields,

that leads to a homogeneous Helmholtz equation in terms of electric-field component E_z . The tangential component of the magnetic field at each connection ($\rho = R$) is equivalent to the magnetic field in the corresponding microstrip access line to meet the boundary conditions. Therefore the tangential component of the magnetic field is constant over the microstrip width access and is zero elsewhere (Fig. 4.2).

$$H_\phi(R, \phi) = \begin{cases} a & -\frac{\pi}{3} - \psi < \phi < -\frac{\pi}{3} + \psi \\ b & \frac{\pi}{3} - \psi < \phi < \frac{\pi}{3} + \psi \\ c & \pi - \psi < \phi < \pi + \psi \\ 0 & \text{elsewhere,} \end{cases} \quad (4.7)$$

where $\psi = \sin^{-1}(w/(2R))$, with w the width of the microstrip lines in the accesses.

Hence, a Green's function can be introduced to describe the azimuthal electrical component such that

$$E_z(\rho, \phi) = \int_{-\pi}^{\pi} G(\rho, \phi, R, \phi') H_\phi(R, \phi') d\phi', \quad (4.8)$$

where $G(\rho, \phi, R, \phi')$ is given in [56].

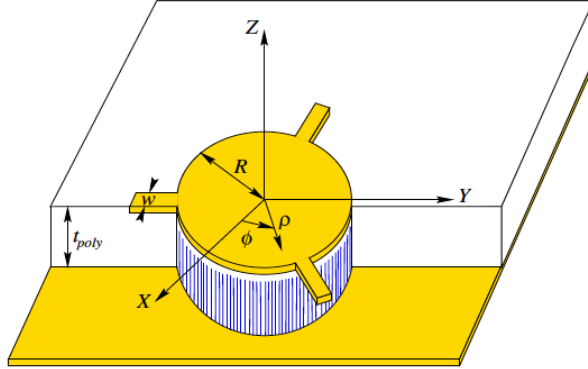


Figure 4.2 – Sketch of microwave circulator based on an unbiased ferromagnetic nanowire array embedded in porous template, showing the ground plane and microstrip pattern deposited on the substrate [36].

4.3.3 Circulation mechanism and field distribution

For this model, we only consider a magnetic wall at the edge of the circulator disc. Those resonance occur if the denominator of the series involved in the Green's function is zero as follows :

$$J'_n(kR) - \frac{\kappa}{\mu} \frac{n J_n(kR)}{kR} = 0, \quad (4.9)$$

where J is the Bessel function of the first kind, n may be any positive or negative integer and denotes the order of the resonant modes, k is the effective propagation constant defined by

$$k = \omega \sqrt{\frac{\epsilon_0 \mu_0 \epsilon_{eff} (\mu^2 - \kappa^2)}{\mu}}. \quad (4.10)$$

The solutions for a number of resonant modes up to 17 have been computed thanks to numerical simulations. For each value of n , their exist values of kR that respect the resonance in eq. (4.9). When the substrate below the disc is isotropic, $\kappa/\mu = 0$, the resonant conditions of the clockwise and counterclockwise rotating modes are identical. If an anisotropy appears, $\kappa/\mu \neq 0$, like in our unbiased nanowired substrates, each degenerate pair $(n, -n)$ splits, giving different solutions of eq. (4.9): at fixed radius R , these two resonances have different frequencies. The circulation frequency is a specific situation, situated between these two resonant frequencies, where the rotating modes travel at different velocities, which implies that one rotating mode have twice the speed of the other one.

The electric field E_z normal to the alumina membrane, is derived from (4.8), where the Green's function $G(\rho, \phi, R, \phi')$ is integrated over the disc circulator circumference, and the magnetic field at the edge $H_\phi(R, \phi)$ is given by (4.7). The corresponding azimuthal magnetic field H_ϕ is then derived from E_z using Maxwell's equations.

$$E_z(r, \phi) = \sum_{n=-\infty}^{+\infty} a_n J_n(kr) e^{jn\phi}, \quad (4.11)$$

$$H_\phi(r, \phi) = \sum_{n=-\infty}^{+\infty} j \frac{a_n}{\zeta_{eff}} \left[J'_n(kr) - \frac{\kappa}{\mu} \frac{n J_n(kR)}{kR} \right] e^{jn\phi}, \quad (4.12)$$

where J_n is the n^{th} -order Bessel function, r the radial position, ϕ the azimuthal angle and k the effective propagation constant, while

$$\zeta_{eff} = \left(\frac{\mu_0 (\mu^2 - \kappa^2)}{\epsilon_0 \epsilon_{eff} \mu} \right)^{\frac{1}{2}} \quad (4.13)$$

is the intrinsic wave impedance. The effective permittivity and permeability are present in the equations defining the electric and magnetic

field inside the circulator. Both influence the resolution of the Bessel function. Another point to signal is the ratio of the permeability components κ/μ that give the change for different resonant solutions. The normalized magnetization $m = M/M_s$ must not be zero to satisfy the degenerescence of the resonance solution, therefore the nonreciprocal effect of the present device.

4.3.4 Matching network

The input impedance Z_{in} at the connection of the input port can be calculated from the impedance matrix subjected to the circulation condition. An approximation allows to find the impedance matching: the transmission occurs from ports 1 to 2, port 3 being isolated. The three-ports circulator is then reduced to two ports

$$\begin{cases} V_1 = Z_{11}I_1 + Z_{12}I_2, \\ V_2 = Z_{21}I_1 + Z_{22}I_2, \end{cases} \quad (4.14)$$

with the symmetrical geometry of the circulator, the following matching condition holds:

$$Z_{in} = \frac{V_1}{I_1} = -\frac{V_2}{I_2}. \quad (4.15)$$

V_i and I_i , $i = \{1, 2\}$, are the voltage and the current at the connections between microstrip access lines and circulator disc for both ports. It can be calculated thanks to (4.11) and (4.12) taking $r = R$ and $\phi = \pm\pi$. After some calculations and considering $Z_{11} = Z_{22}$ due to the symmetry, the following expression is found:

$$Z_{in\ 1,2} = \pm \sqrt{Z_{11}^2 - Z_{12}Z_{21}}. \quad (4.16)$$

In order to reduce the insertion losses, the input impedance has to be matched to the 50 Ω -impedance of the VNA. This matching is divided in two parts, which can be solved separately by calculating the size of geometrical design (Fig. 4.3). The first part is to get a 50 Ω -impedance at the connection of the access lines of the circulator. The formula that calculates the width of the microstrip line to obtain $Z = 50\Omega$ can be used [33]:

$$W_{out} = \frac{377h_p}{\sqrt{\epsilon_{eff}Z}}, \quad (4.17)$$

where h_p and ϵ_{eff} are the height of the alumina membrane and the effective permittivity of the substrate, which is the permittivity of the

nanoporous alumina membrane in this case where no nanowire is below the access lines. The length of the access line L has to be the quarter of the wavelength at the circulation frequency. To avoid the reflection to the entries of the circulator disc, the width of the access lines at the entry of the circulator disc W_{in} is determined by iterative program to satisfy this following condition:

$$Z = \frac{Z_c \cdot Z_c}{Z_{in}} = 50\Omega. \quad (4.18)$$

To improve the performances of the circulator, we should match the ports at the circulation frequency of the device. This circulation frequency is weakly dependent on the size of the access to the disc, we should calculate the widths and length thanks to iterative simulations that converge the values of dimensions to the 50Ω -matching at the circulation frequency.

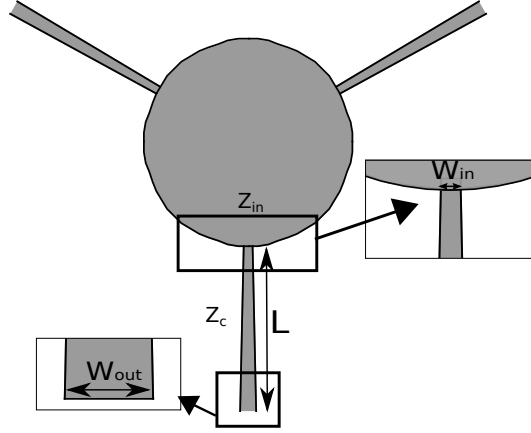


Figure 4.3 – Different dimensions that have to be adapted to match the 50Ω -impedance to reduce the insertion losses due to the reflection. W_{out} is the width of the access lines at the connections with the VNA, L is the length of the access lines and W_{in} is the width of the access lines at the entry of the circulator disc.

This formula will be used in the numerical optimization of the circulator working in the X-band to achieve the required standard specifications.

4.4 Experimental dependence of the circulation frequency to the nanowires length

Four microwave circulators based on NiFe ferromagnetic nanowire arrays with different lengths of nanowires were fabricated. Each of them has shown circulations in two distinct frequency ranges, around 11 GHz and 25 GHz, respectively. The FMR frequency of the NiFe nanowired substrate was previously measured and found to be at 12.5 GHz. In Fig. 4.4 (left hand side), we can see the measured (red) and simulated (blue) forward and backward transmission for a 3 mm circulator based on a 56%-relative length nanowired substrate in the two frequency ranges. There is a good agreement in the determination of circulation frequencies and their amplitudes between modeled and experimental results.

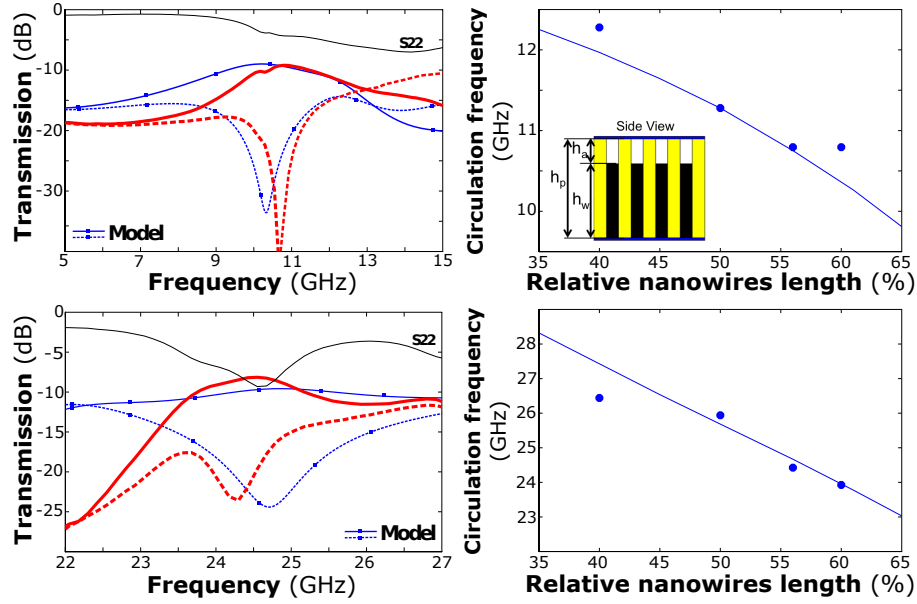


Figure 4.4 – (left) Measured (red) and predicted (blue-squared) forward and backward transmissions for a circulator based on the same ferromagnetic NiFe nanowire array with 56%-relative length, at two different frequency ranges. The measured S_{22} reflections (black lines) is given for information. (Right) Dependence of the circulation frequencies on the length of nanowires for predicted (lines) and measured (dots) results for the same circulator, at different frequency ranges.

To demonstrate the dependence of the circulation frequencies with the length of the nanowires, the right hand side of Fig. 4.4 shows the

circulation frequencies respectively extracted from S -parameters measured (dots) on the devices based on substrate with various lengths of nanowires (for 40%, 50%, 56% and 60%), and the predicted (lines) from the simulations using the same filling range.

We experimentally showed the dependence of the non-reciprocal effect with the variation of the nanowires length that allows fine-tuning of the working frequency of the device. The good agreement between simulations and experiments suggests that the model can be used to predict with good accuracy the microwave behavior of the circulator.

4.5 Microwave circulator in the X -band for airborne applications

This section is linked to the project in collaboration with *Thalès Systèmes Aéroportés* and its purpose is to design and fabricate a microwave circulator based on an unbiased magnetic nanowire array embedded in a nanoporous alumina membrane, which has the working frequency in the X -band (8-12 GHz). The required specifications to industrially develop this kind of application are an isolation greater than 15 dB and insertion losses lower than 2 dB. We first selected the magnetic nature of nanowires, then we proceeded to a series of numerical simulations that define the geometrical properties of the nanowired substrate where a circulation appears in the X -band. We then designed and realized some devices by the electroplating and the laser micromachining techniques. We finished with a numerical optimization of the parameters to get the required specifications.

4.5.1 Ferromagnetic alloys for the nanowired substrate

The choice of the used magnetic material for the nanowires has been done in the range of the materials or alloys previously studied in our group, namely Ni or Co, and the alloys NiFe or CoFe. We decided to exclude the nickel material that presents an additional magnetic contribution in small diameter alumina membrane. Cobalt material has been rejected due to its high magneto-cristalline component when using an electrolytic solution with usual pH. The properties of NiFe and CoFe alloys are well known, and in addition the saturation magnetization is quite different between both, which gives distinct ferromagnetic resonance frequencies.

4.5.2 Experimental confirmation

We first determined a range of geometric parameters which provide a circulation frequency in the X -band with the required specification, as the diameter of the circle, the porosity of the membrane and the length of nanowires. Table 4.1 shows all the experimental samples that have been fabricated

	Name	φ	h_p	P	h_w	D	f_{circ}
NiFe	Py1	35 nm	100 μm	12 %	40 %	3 mm	10.9 GHz
	Py2	35 nm	100 μm	12 %	50 %	3 mm	10.9 GHz
	Py3	35 nm	100 μm	12 %	56 %	3 mm	11.4 GHz
	Py4	35 nm	100 μm	12 %	60 %	3 mm	12.3 GHz
CoFe	CoFe1	35 nm	100 μm	12 %	50 %	4 mm	<i>No circ</i>
	CoFe2	35 nm	100 μm	12 %	60 %	4 mm	<i>No circ</i>
	CoFe3	18 nm	50 μm	10 %	62 %	3 mm	12.5 GHz

Table 4.1 – Parameters of the microwave circulator working in the X -band.

100 μm -thick and 35 nm-pores diameter *Synkera* membranes were used with the purpose to enhance the matching to 50 Ω -impedance of the device, compared with the 50 μm -thick membrane. The porosity is fixed to 12 %. According to numerical predictions, a 3 mm-diameter circulator has been fabricated in the case of NiFe nanowire arrays and a 4 mm-diameter circulator for CoFe nanowire arrays with a height of 100 μm . As we can see in the table 4.1 and in the Fig. 4.5 (a), circulation frequencies are found in (near to) the X -band in the case of NiFe nanowires, with high isolation (> 30 dB). In the case of CoFe nanowires, no circulation is seen for the samples in the 100 μm -thick membranes as the remanence of these magnetic arrays is low. Too high porosity membrane and poor quality of the membrane could explain this drop of the remanence. A FMR characterization informs that the effective porosity of the CoFe nanowire arrays seems to be at 15 %, instead of 12 %.

As demonstrated in section 2.3, CoFe nanowire arrays embedded in AAO membrane with pores diameter $\varphi = 35$ nm show low magnetic remanence, which could explain the lack of the nonreciprocal circulation. Smaller pore diameter and porosity membranes enhance the remanence of the CoFe nanowire arrays.

To avoid the drop of the remanence in CoFe nanowire array, we used an 18 nm-pores diameter and 50 μm -thick AAO membrane with porosity $P = 10$ %. The remanence of this kind of sample is high thanks to a lower porosity but also to the small nanowires diameter and an nonreciprocal

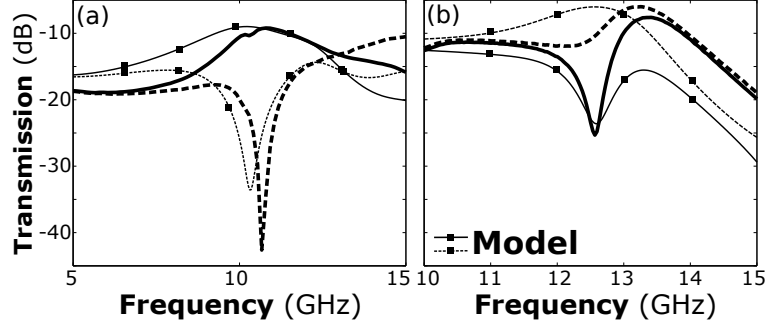


Figure 4.5 – Frequency spectra of microwave circulators based on (a) NiFe nanowire arrays embedded in a 100 μm -thick and 35 nm-pores diameter AAO membrane with porosity of 12 % and nanowires length 56 %; and (b) CoFe nanowire arrays embedded in a 50 μm -thick and 18 nm-pores diameter AAO membrane with porosity 10 % and nanowires length 62 %. Thin squared curves are the numerical simulations with the same geometric parameters as samples.

effect is seen in the form of a circulation near the *X*-band (Fig. 4.5 (b)). Insertion losses at the circulation frequency are high in either NiFe or CoFe samples due to high dielectric losses of the alumina membrane. The loss tangent $\tan \delta$ has been predicted thanks to the model to be 0.04 although a value of 0.001 could be reached for porous alumina [94]. As explained in section 2.2, dielectric losses is enhanced by the porosity of alumina and the presence of H_2O molecules inside the pores of the membrane [93]. The ferromagnetic resonance of NiFe nanowires also contributes to the high insertion losses for this kind of samples.

4.5.3 Numerical optimization

To improve the transmission at the circulation frequency to match with the industrial specifications (< 2 dB), a numerical characterization is done for CoFe nanowire arrays with ideal dielectric losses of porous alumina membrane ($\tan \delta = 0.001$) and with matched ports to 50 Ω (see Fig. 4.3). Fig. 4.6 shows numerical predictions for three configurations of CoFe nanowire circulators. The first one (red curves) is done with high dielectric loss membrane ($\tan \delta = 0.04$) and straight (mismatched) access line geometry ($W_{\text{out}} = W_{\text{in}} = 500 \mu\text{m}$, $L = 100$ mm). The second one considers also $\tan \delta = 0.04$, but tapered quarter wavelength 50 Ω -matching structure as access line ($W_{\text{out}} = 280 \mu\text{m}$, $W_{\text{in}} = 182 \mu\text{m}$, $L = 3.192$ mm). The third one is the fully optimized case combining a low

loss substrate $\tan \delta = 0.001$, and the 50Ω -matching structures. Those numerical results demonstrate that the insertion loss can be reduced to less than 2 dB while preserving a good value of isolation (15 dB).

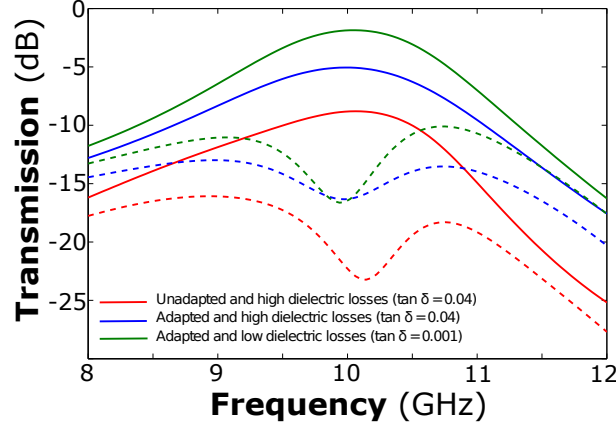


Figure 4.6 – Predicted transmission of a circulator operating at frequencies close to 10 GHz, based on a CoFe nanowire array with 50 % relative nanowires length for different cases: using 50Ω -mismatched ports and high dielectric losses AAO membrane ($\tan \delta = 0.04$) (red curves), 50Ω matched ports and high dielectric losses AAO membrane ($\tan \delta = 0.04$) (blue curves) and 50Ω -matched ports and low dielectric losses AAO membrane ($\tan \delta = 0.001$) (green curves).

4.6 Two-ports isolator based on three-ports junction topology for aerospace applications

The microwave circulator is a three-ports application that serves to route signal to the right direction, i.e. from the transmitter to the antenna and from the antenna to the receiver. A specific configuration, which applies a short-circuit to the third port, transforms this circulator in a two-ports device that can be used as an isolator. This section is linked to the project done in collaboration with *Thalès Alenia Space* (see section 3.6) with the purpose to conceive a microwave isolator based on the unbiased ferromagnetic nanowire array embedded in an alumina membrane for the aerospace applications, more specifically, to find a device with an isolation in the required frequencies ranges: *Ku* band for emission (17.3 - 20.2) GHz and reception (27 - 30) GHz, and *Q, V* band for emission (37.5 - 42.5) GHz and reception (47.2 - 50.2) GHz.

4.6.1 Numerical parametric study

For the modeling, the 3×3 matrix transmission of the circulator becomes a 2×2 matrix. The numerical program is the same as the microwave circulator, excepted the condition to cancel the third port, meaning that the in the 3×3 -matrix impedance, the third column and the third line are considered as zero. We may use the same numerical program to find all the parameters leading to obtain an isolation higher than 15 dB in the two required frequency ranges.

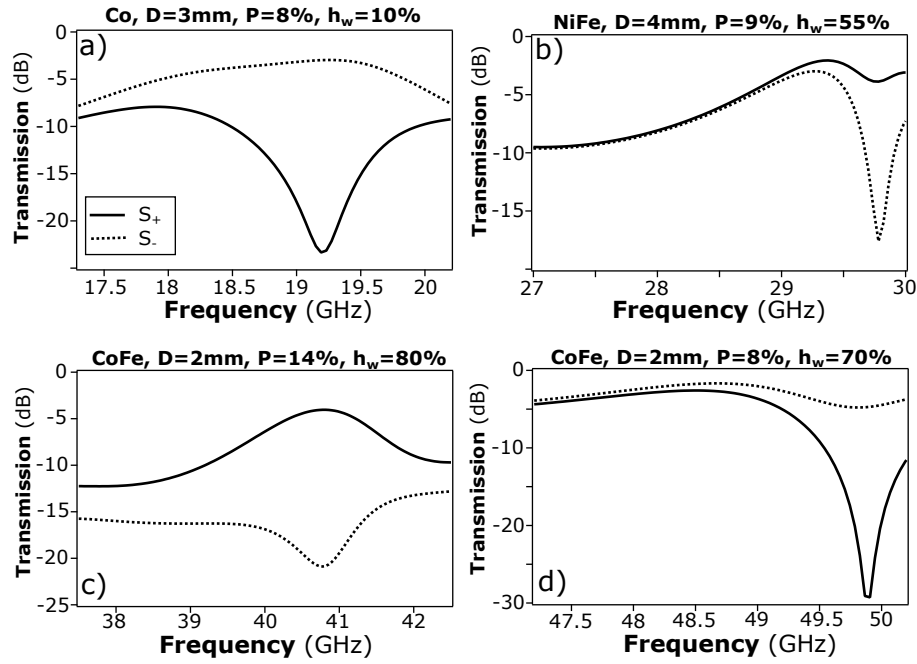


Figure 4.7 – Numerical forward S_+ and backward S_- transmission of the three-ports junction isolator based on the microwave circulator device for the required frequency bands. The used parameters are shown above each graph.

The height of the membrane is fixed to $90 \mu\text{m}$, the dielectric constant to 9.8 and the loss tangent to 0.001, which corresponds to the ideal case. Some numerical simulations that take into account all the variables (packing factor, diameter of the disc, length of nanowires or magnetic nature of the nanowires) allowed fixing a few parameters for each desired frequency range that show an isolation of more than 15 dB and with insertion losses less than 5 dB. In Fig. 4.7, the best numerical results for each frequency band are shown and the geometric parameters as well the

nature of nanowires is written: D is the disc diameter, P is the packing density and h_w is the relative length of nanowires. Nonreciprocal effect, given by the non-diagonal term of the permeability tensor, is ensured by the large volume of magnetic matter in the case (b), (c) and (d) and by the proximity of the FMR frequency in the case (a).

An optimization of the transmission can be done, keeping the same parameters, but just increasing the height of the membrane that improves the matching of the transmission line to 50Ω -impedance. The table 4.2 gives the values for numerical optimization for higher membranes. An improvement up to 2.2 dB is observed for the 47.2 to 50.2 GHz frequencies range, just increasing the membrane height to $400 \mu\text{m}$.

Frequency bands (GHz) and nanowires nature	Working frequency (GHz)	Isolation (dB)	Insertion losses (dB) (for $h_p = 90 \mu\text{m}$)	Insertion losses (dB) (optimization)
17.3 - 20.2 Cobalt	19.1	20	3	2.3 for $h_p=200\mu\text{m}$
27 - 30 NiFe	29.7	14	3.9	2.6 for $h_p=200\mu\text{m}$
37.5 - 42.5 CoFe	40.8	17	4	3.5 for $h_p=200\mu\text{m}$
47.2 - 50.2 CoFe	49.8	25	4.7	2.5 for $h_p=400\mu\text{m}$

Table 4.2 – Best numerical results and parameters from 3-ports isolators for each frequencies band.

Another way exists to minimize the insertion losses, by avoiding the reflection in the access transmission lines or in the disc. The dimensions of the access lines (W_{in} , W_{out} and L , see Fig. 4.3) can be calculated to match the 50Ω -impedance of the VNA at the circulation frequency. L must equalize the quarter of the wavelength, W_{out} is defined by the eq. (4.17) and W_{in} must verify the eq. (4.18). Because of the dependence of the working frequency to the size of the access lines, this value could be found using an iterative program, as used for the microwave circulator. The convergence of this value meets some problems due to the specific configuration, with the short-circuit to the third port, which has consequences on the stability of the program.

However, we remain optimistic that an adaptation of the size of the ac-

cess transmission lines, which could be coupled with an increase of the membrane height, will give a final optimization to reach the required specifications.

4.6.2 Experimental confirmation

Isolators based on the three-ports configuration, with a short-circuit on the third port, were fabricated with NiFe and CoFe nanowired substrates to prove the feasibility of the concept. Porous alumina membranes from *Synkera Technologies* are used for the fabrication. These membranes have different geometrical properties according to the nanowires nature: for the NiFe nanowired substrate, we used nanoporous alumina with pores diameter 35 nm, porosity 12 % and thickness 100 μm , while for the CoFe nanoporous alumina with pores diameter 18 nm, porosity 10 % and thickness 50 μm is used. The reason of this change aims to keep a high remanence for both NiFe and CoFe magnetic arrays.

Table 4.3 gives all the fabricated samples and their parameters. NiFe and CoFe isolators fabrication are based on the parameters of the microwave NiFe and CoFe circulators (table 4.1). The diameter of the disc is 3 mm and the accesses transmission lines have a length of 3 mm and a width of 200 μm . The short-circuit is made by depositing a gold layer at the end of the third port, which links the transmission access line of the device to the ground (Fig. 4.8).

	Name	φ	h_p	P	h_w	D	f_{isol}
NiFe	Py2	35 nm	100 μm	12 %	50 %	3 mm	16 GHz 27 GHz
	Py3	35 nm	100 μm	12 %	56 %	3 mm	11 GHz 25 GHz
	Py4	35 nm	100 μm	12 %	60 %	3 mm	11 GHz 24 GHz
CoFe	CoFe1	18 nm	50 μm	10 %	65 %	3 mm	13 GHz 21 GHz 40 GHz
	CoFe2	18 nm	50 μm	10 %	60 %	3 mm	18 GHz 27 GHz

Table 4.3 – Parameters of the 3-ports configuration isolators.

Fig. 4.9 shows the spectra for three desired frequency bands required in the conception of spatial microwave applications for various 3-ports isolators based on (a) a CoFe nanowires array with $h_w = 60$ %-relative

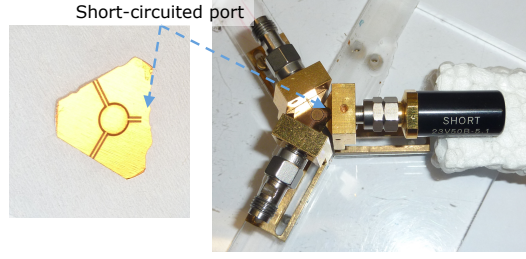


Figure 4.8 – Pictures of the isolator in three-ports configuration with the short-circuit in the third port symbolized by the link between the transmission access line and the ground of the device. Short-circuited item is connected to ensure good leakage of the microwave signal to the ground.

nanowires length into an AAO membrane with membrane height $h_p = 50 \mu\text{m}$, pores diameter $\varphi = 18 \text{ nm}$ and porosity $P = 10 \%$ where we observe an isolation larger than 25 dB at 18.2 GHz with high insertion losses 11 dB; (b) a NiFe nanowire isolator into $h_w = 50 \%$ -relative nanowires length into an AAO membrane with membrane height $h_p = 100 \mu\text{m}$, pores diameter $\varphi = 35 \text{ nm}$ and porosity $P = 12 \%$, where we observe an isolation about 16 dB at 27.5 GHz and insertion losses about 18 dB; and (c) a CoFe nanowire isolator with $h_w = 65 \%$ -relative nanowires length into an AAO membrane with membrane height $h_p = 50 \mu\text{m}$, pores diameter $\varphi = 18 \text{ nm}$ and porosity $P = 10 \%$ where the isolation is situated at 40.3 GHz with isolation value 20 dB and high insertion losses about 17 dB.

The isolation values of each device meet the required specification ($> 15 \text{ dB}$), however insertion losses are still very high to develop industrially some kind of microwave applications. Insertion losses observed in both CoFe nanowire devices have a value higher than 10 dB (Fig. 4.9(a)(c)), while losses in the NiFe 3-ports isolator reaches 15 dB (Fig. 4.9(b)). If high insertion losses are mainly the consequence of high dielectric losses of the commercial AAO membrane, as explained in chapter 2.2, the transmission level in the three-ports junction topology is also the consequence of rotating modes. Magnetic losses due to the FMR absorption can be neglected since the FMR frequencies of CoFe or NiFe nanowires stay far from working frequencies: 12.3 GHz for NiFe nanowires (Fig. 4.9(b)) and 25.2 GHz for CoFe nanowires (Fig. 4.9(a)(c)). The frequency dependence of the dielectric losses can explain the larger insertion losses at high frequency range (Fig. 4.9(c)) compared to Fig. 4.9(a). High losses observed in the NiFe isolator (Fig. 4.9(b)) are due to

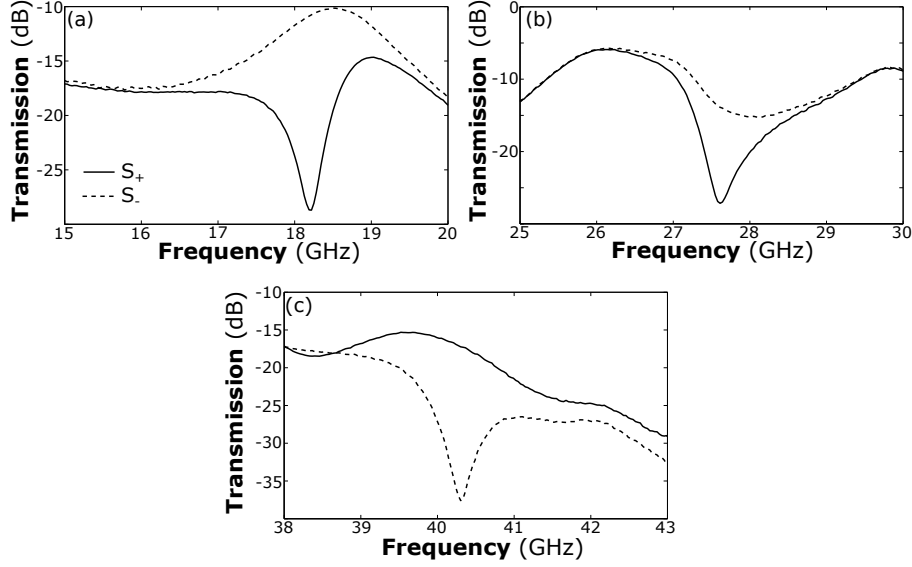


Figure 4.9 – Frequency spectra of three-ports configuration device for (a) CoFe nanowire isolator with $h_w = 60$ %-relative nanowires length into a alumina membrane with membrane height $h_p = 50 \mu\text{m}$, pores diameter $\varphi = 18 \text{ nm}$ and porosity $P = 10 \%$, (b) NiFe nanowire isolator into $h_w = 50$ %-relative nanowires length into AAO template with membrane height $h_p = 100 \mu\text{m}$, pores diameter $\varphi = 35 \text{ nm}$ and porosity $P = 12 \%$, (c) CoFe nanowire isolator with $h_w = 65$ %-relative nanowires length into AAO template with membrane height $h_p = 50 \mu\text{m}$, pores diameter $\varphi = 18 \text{ nm}$ and porosity $P = 10 \%$.

high dielectric losses of the AAO membrane but also the consequence of non-completely full rotating modes below the disc of isolator.

Larger membrane heights theoretically yield better transmission, as shown in table 4.2 and as observed in Fig. 4.10 showing the transmission spectra for NiFe nanowire circulator with $h_p = 100 \mu\text{m}$. We report on this device a microwave isolation at $f = 16.3 \text{ GHz}$, near the 17.3-20.2 GHz frequency range, with isolation value 16 dB and insertion loss 4 dB. The lower insertion losses observed for this NiFe sample compared to the CoFe sample (Fig. 4.9(a)) could be explained by the increase of the substrate height from $50 \mu\text{m}$ to $100 \mu\text{m}$.

Thanks to numerical predictions and the experimental evidences, we demonstrated the conception and fabrication of microwave isolators based on 3-ports junctions with nanowired substrate. The high unbiased working frequencies and planar fabrication would allow full integration of such devices inside a micro-electronic circuit for space applications.

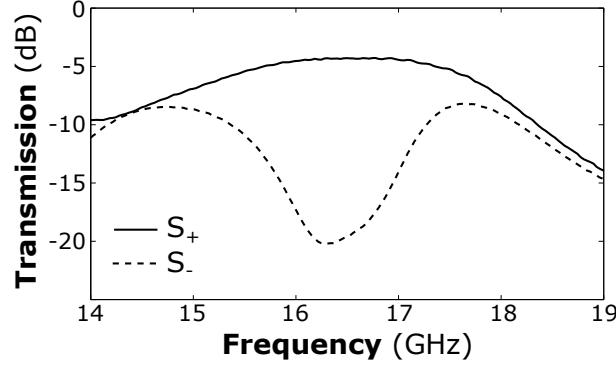


Figure 4.10 – Frequency spectrum of a three-ports configuration device for NiFe nanowire isolator of $h_w = 50$ %-relative nanowires length into AAO template with membrane height $h_p = 100 \mu\text{m}$.

4.7 Conclusions

Ferromagnetic nanowire arrays have been used to conceive and fabricate planar microwave circulators and isolators based on the specific three-ports junction configuration. Working frequencies of these devices allow to dedicate them to airplane applications for X -band microwave circulators or to aerospace applications for Ka - and Q, V -bands isolators.

We investigated the dependence of the microwave behavior to the nanowires length with the fabrication of four circulators based on NiFe nanowire arrays having different nanowires lengths. Theoretical model developed in the specific case of magnetic nanowires confirms the behavior of circulator to nanowires length and numerical simulations predicts the dependence of working frequencies to these geometric parameters. The experimental confirmation permits to conceive and to tune circulators with precise working frequencies. Further numerical characterizations should be done to forecast the magnitude or the linewidth of isolation for specific circulation to geometric parameters. NiFe and CoFe nanowire circulators for airborne applications show circulation with high isolation and reproducibility in (or near to) the X -band. The magnetic remanence was an issue for $100 \mu\text{m}$ -thick CoFe nanowire circulator for which the remanence value was too low to provide nonreciprocal effect to the device. The bad quality of commercial AAO membranes coupled to the high saturation magnetization for CoFe have driven to this drop of magnetic remanence. The use of lower diameter and porosity membranes solved this problem. High insertion losses are observed in the different experimental measurements as usual for commercial AAO

membranes where high dielectric losses should explain the low transmission value. In both NiFe and CoFe numerical predictions, value of $\tan \delta$ is fixed to 0.04 to agree with experimental results, while an ideal value of $\tan \delta = 10^{-3}$ is attainable for 10 %-porous alumina. Another possibility to improve the transmission is to reduce the reflection in the device by adapting the size of its accesses to 50Ω -impedance and quarter wavelength. An iterative program allows calculating precisely the widths and the length of transmission lines accesses and the new fabrication method for drawing the design of circulator using the laser micromachining system gives a chance to precisely adapt these numerical parameters to the experimental devices. Numerical resulting performances of this improved case meet the commercial standard requirements and confirm the high potential of the nanowired substrates for on-board systems in aerospace applications.

The conception of microwave isolators based on the similar configuration shows the wide existing option panels from magnetic nanowire arrays. Transforming the 3-ports circulators to a 2-ports isolators by applying a short-circuit to the third port gives other possibilities for airborne and aerospace applications. Numerical characterizations allow finding with several geometric or nanowires nature variables an isolation in each frequencies band used in aerospace: Ka - or Q, V -bands for reception and emission. Experimental confirmations show the simplicity and the reproducibility of the fabrication although the numerical predictions does not agree completely to the experimental measurements due to the high sensitivity of the results to the short-circuit. Once again, high dielectric losses of the AAO membrane are the issue in the way to develop industrially these kinds of devices. So, a significant improvement needs to be done on the fabrication of AAO membranes in order to reduce their dielectric losses. The development of the solutions cited in the section 2.2 may be applied for this application.

Chapter 5

Ferromagnetic inductors

5.1 Introduction

The microelectronics industry is in demand for compact integrated inductors with high inductance and quality factor operating up to several GHz. A first solution has been developed using a low-loss substrate, like bulk alumina [57] or porous silicon [58] allowing a decreasing of losses of the system. A second solution is to include a ferromagnetic core in the structure to increase the inductance value, but with the main drawback that FMR losses and the eddy current induced in the volume of the core deteriorate the quality factor at *rf* frequency. Using as substrate a high aspect ratio nanowire array embedded into a nanoporous alumina membrane combines both solutions : the high porosity and thickness of the self-supported membrane enable to supply a large amount of magnetic material in order to maximize the value of the inductance while the nanowired alumina membrane is also able to drastically limit substrate losses up to several GHz and avoid the eddy currents in the ferromagnetic material, thanks to the nanoscale of the wires.

Through experiments, we demonstrated the feasibility of using the standards machining from microelectronics to fabricate microwaves devices based on ferromagnetic nanowires inside self-supported nanoporous alumina membrane, leading to a possible integration for systems in package (SiP) or on chip (SoC). We compared various inductor geometries based on two kinds of substrates: a self-supported AAO membrane with or without Ni nanowires. We observed a simultaneous increase of inductance and quality factor up to 7 GHz. A theoretical model, which includes geometrical and material parameters, validated the experiments. We also used this model to optimize the enhancement of those two parameters by tuning the porosity or the filling height of the pores.

5.2 Topology and operation

The filling of the membrane with the nanowires is carried out in one step with the purpose of obtaining one large circular zone where Ni nanowires are grown. Large porosity $P = 35\%$ and thickness $h_p = 60\ \mu\text{m}$ allow having an important volume of ferromagnetic material in order to enhance the FMR effect. The nanowires length has 40 % ratio of the membrane height. A large number of inductor designs (Fig. 5.1), including meander and spiral inductors, is etched by optical lithography technique in the Al layer previously deposited onto the side of the membrane opposite to the one with the evaporated cathode.

All the process of fabrication can be found in section 2.1, i.e. the electrolytic solutions parameters listed in table 2.1. The list of sample used in this chapter and its geometric parameters, as well as the fabrication technique for the transmission line, can be found in table 2.2.

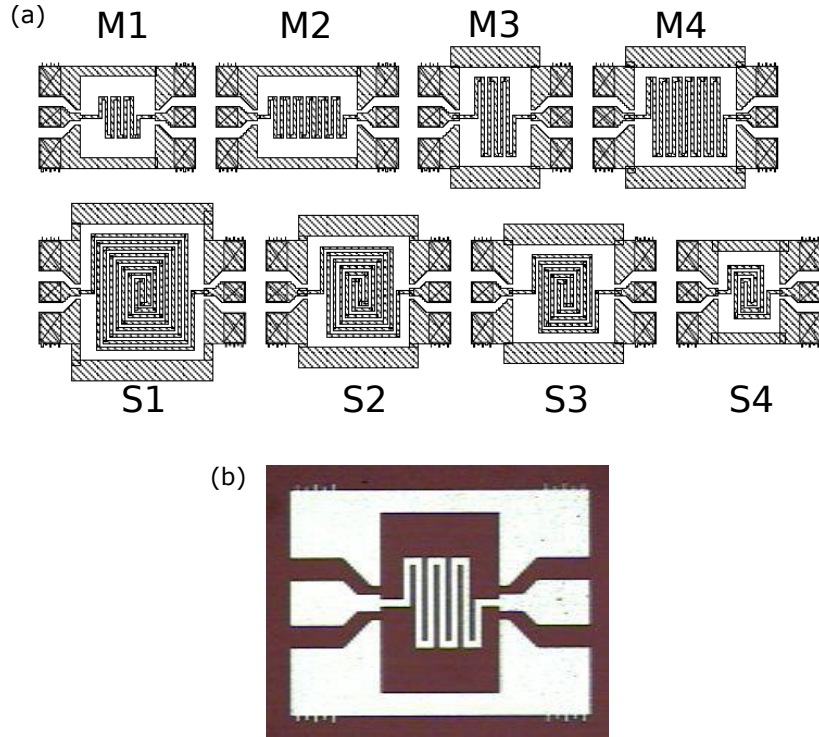


Figure 5.1 – (a) Figure of planar inductors from the mask used for the lithography. The first line represents the meander inductors, and the second line, the spiral inductors. (b) SEM image from the M1c planar inductor.

Simulations are performed using an analytical model [109, 110] for frequencies from 0.5 GHz to 15 GHz. Calculations consider the ferromagnetic resonance of nanowires involved in the formula of relative permeability of the substrate, as explained in section 2.2, dielectric losses of alumina membrane depending on porosity, the coupling between strips of the inductors and the skin depth effect from aluminum electrodes. Simulations calculate the S -matrix characterizing the propagation of signal between ports and its reflection at each port.

The planar inductors are divided in two classes depending on their geometry : the meander M and the spiral S groups (Fig. 5.1). The actual geometry of the meander and the spiral are: width and thickness of strips equal to $16\ \mu\text{m}$ and $500\ \text{nm}$, respectively, spacing between strips equal to 3, 7, 10 or $15\ \mu\text{m}$. The vertical lengths of meander inductors are $200\ \mu\text{m}$ and $400\ \mu\text{m}$, and the smallest vertical length of spiral inductor is $130\ \mu\text{m}$.

5.3 Analytical model

Thanks to the equivalent circuit defined on Fig. 5.2, we can calculate the Y -parameters from S -matrix values, and extract all elements to show their rf dependence, as defined in [65]. The inductance L and the resistance R correspond to the planar inductor and depend on the magnetic behavior of the substrate and the ohmic losses of stripline conductor, respectively. Conductances $G_{1,2}$ and capacitances $C_{1,2}$ are directly issued from the dielectric behavior of the substrate and represent impedances between ground and port 1 or 2. Owing the geometrical symmetry of planar inductor, we consider $G_1 = G_2 = G$ and $C_1 = C_2 = C$. Quality factor is found using complex impedances : $Q = 2\pi f \frac{L+C}{R+G}$. In our case, capacitance and conductance values are negligible compared respectively to inductance and resistance values, so the quality factor rewrites $Q = 2\pi f \frac{L}{R}$.

5.4 Results and discussion

A dozen of inductors has been measured and a comparison with and without nanowires can be done. All the measured planar inductors are listed in the table 5.1.

In the first part of this section, we show and discuss the results of the M1c inductor. It is the best typical results that we obtained for all the measured planar inductors listed in table 5.1. Then a summary with

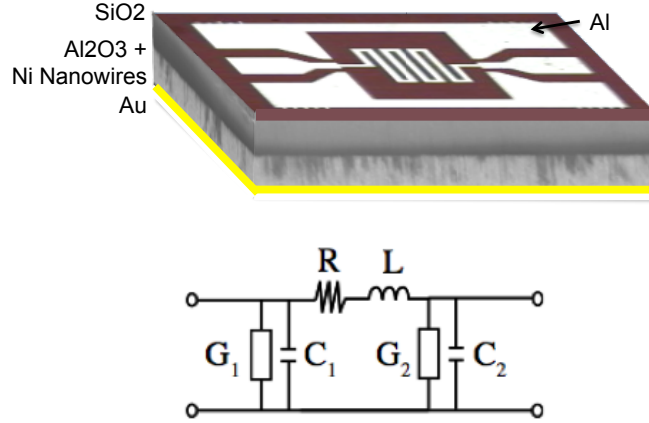


Figure 5.2 – (a) Schematic representation of a planar meander inductor onto a nanowired substrate (b) Equivalent circuit symbolizing the planar inductor on a nanowired substrate.

(a)		M1	M2	M3	M4
Lines interdistance	4 μm	-	M2a	M3a	M4a
	7 μm	M1b	-	M3b	-
	10 μm	M1c	M2c	-	-
	15 μm	M1d	M2d	M3d	-
(b)		S1	S2	S3	S4
Lines interdistance	4 μm	-	S2a	S3a	S4a
	7 μm	-	-	-	-
	10 μm	-	-	S3c	-
	15 μm	-	-	-	S4d

Table 5.1 – Tables of the fabricated (a) meander and (b) spiral planar inductors.

results of all samples is given to compare the different geometries.

5.4.1 M1c planar inductor results

Fig. 5.3 represents the transmission amplitude and phase of the sample number M1c containing Ni nanowires. It is evident that the small local minimum and the inflection observed respectively on the magnitude and the phase at 9 GHz are the signature of ferromagnetic resonance of

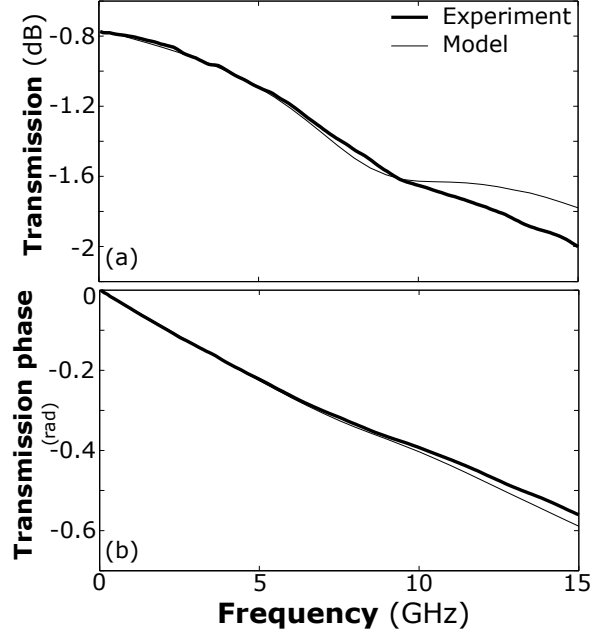


Figure 5.3 – (a) Magnitude and (b) phase transmission of the M1c meander inductor on a Ni nanowired substrate.

the nickel nanowire array. This frequency corresponds to the calculated FMR frequency using high porosity model, $f_r = \frac{1}{2}\gamma M_s$ where $\gamma = 3.09$ GHz·kOe⁻¹ and $M_s = 485$ emu·cm⁻³ are the gyromagnetic factor and the saturation magnetization of Ni, respectively [111].

Effective permeability

The high porosity of the nanowire array and the high nanowires diameter changes the definition that we previously wrote in chapter 2.2. Due to the high dipolar coupling, the remanent magnetization m can be defined as zero. The effective permeability of the nanowired substrate depends on the relative permeability of nanowires in the low frequency range [112] and on the FMR. It can be defined as a tensor:

$$\mu_{eff} = \begin{pmatrix} \mu_{r, nws} + \kappa & -i \cdot \eta & 0 \\ i \cdot \eta & \mu_{r, nws} + \kappa & 0 \\ 0 & 0 & \mu_{r, nws} + \kappa \end{pmatrix}, \quad (5.1)$$

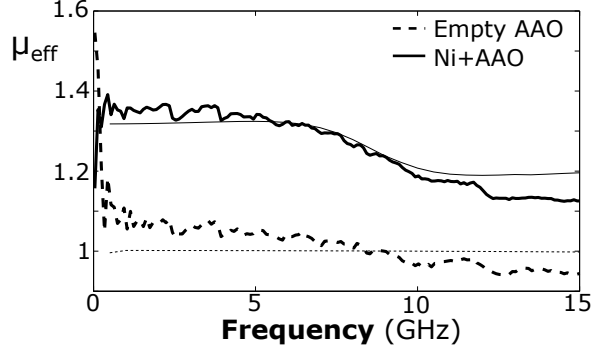


Figure 5.4 – Effective permeability of the nanowired (solid lines) and empty (dashed lines) substrate for the experimental (thick lines) and simulations (thin lines) parts.

with

$$\begin{aligned}\kappa &= 2\pi M_s \gamma P h_w f_r (f_r^2 - f^2)^{-1} (m^2 + 1), \\ \eta &= 4\pi M_s \gamma P h_w f (f_r^2 - f^2)^{-1} m.\end{aligned}\tag{5.2}$$

We could define thanks to the measured S-matrix transmission of the inductors the behavior of the effective permeability from the Ni nanowired and empty substrate. Those observations are in good accordance with the theoretical results obtained by the model above. The effective substrate permeability is larger in the presence of ferromagnetic nanowires, over the whole measured frequency range due to the relative permeability of nanowires, $\mu_{r,nws}$. This increase is even larger below the FMR frequency. We can explain that by the change of sign in the κ term (eq. (5.1)) when frequency f is below or above the FMR frequency f_r . $\Delta\mu_{eff}$ is about +30 % in the frequency range below f_r .

Inductance and resistance values

The behavior of the inductance value is the same as the effective permeability. The increase shown for the permeability affects in the same way the value of the inductance (Fig. 5.5(a)). Indeed, simulations and measurements show that the inductor on nanowired substrate presents a larger value of inductance than the inductor on an empty substrate, and the maximum increase is observed in the frequency range below f_r and is about 30 %. As announced in the introduction, the equivalent resistance of inductor on nanowired substrate shows a maximum at FMR frequency (Fig. 5.5(b)), due to the absorption of microwave by ferromagnetic nanowires. Nevertheless, it is worth noticing that in

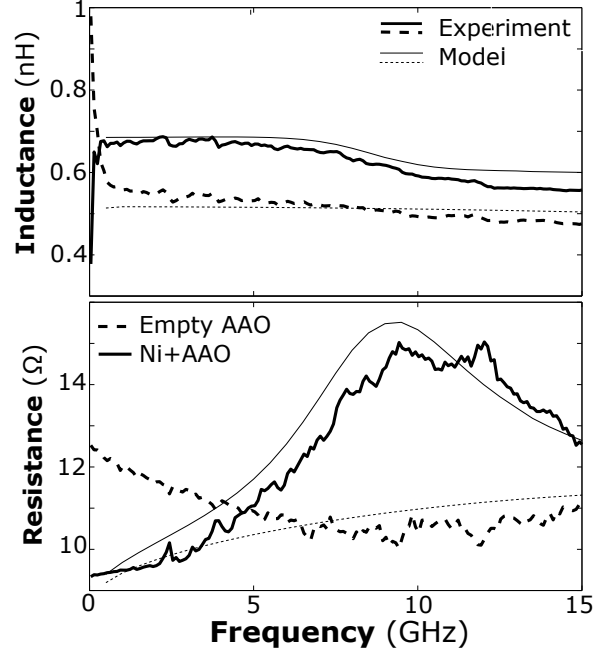


Figure 5.5 – (a) Inductance and (b) resistance values of the inductors M1c in the case of the nanowired (solid lines) and empty (dashed lines) substrate for the experimental (thick lines) and simulations (thin lines) parts.

low frequency range (below f_r), the resistance is of the same order of magnitude ($< 12 \Omega$) for nanowired and empty substrate, because conducting losses in nanowires are minimized even at microwaves due to their nanoscale.

Capacitance value

The behavior of the shunt capacitance induced by the substrate can be modeled by the permittivity model [35] that describes its dependence with the nanowires length. Considering that the bulk alumina permittivity is 9.8, the porosity of the membrane 35% and the Ni nanowires length 40% of the membrane height, the effective permittivity of the substrate is 8.7 and 14.6 for the empty and nanowired cases, respectively. As demonstrated in Fig. 5.6, the shunt capacitances of empty and nanowired substrate are in the same ratio as the effective permittivity.

Quality factor

The consequence of this is an increase of the quality factor in the

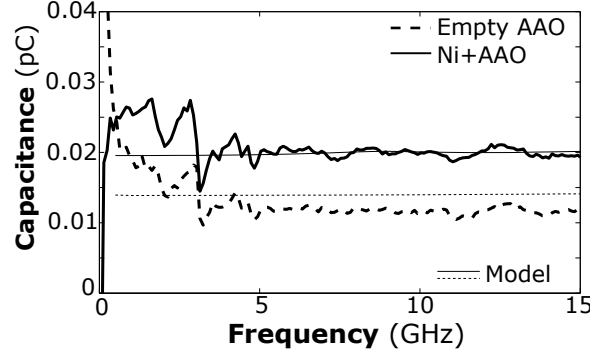


Figure 5.6 – Capacitance value of the inductors M1c in the case of the nano-wired (solid lines) and empty (dashed lines) substrate for the experimental (thick lines) and simulations (thin lines) parts.

frequency range below FMR frequency. Fig. 5.7 shows evidence of this increase for frequencies from 40 MHz to 7 GHz, reaching a maximal value of 23 % at 4 GHz. This value is the largest increase reported in the literature for such ferromagnetic inductors. Reported values are usually lower than 10 % [60], unchanged [61] or even lowered [63, 64]. Simulations predict that the operation frequencies band can be widened to higher frequencies when applying a *dc* magnetic field on the substrate. This magnetic field is parallel to the nanowires and gives rise to a shift to larger FMR frequencies. As before, the inductance value is higher for frequencies below the FMR frequency while losses remain the same for those frequencies. For empty substrate, the *dc* magnetic field does not have any influence because there is no magnetic material. As a result, simulations without and with applied magnetic *dc* magnetic field of 4 and 6 kOe show in Fig. 5.7 an improvement of quality factor higher for frequencies up to 7 GHz, 9 GHz and 14 GHz, respectively. FMR frequencies for those simulations are, respectively, 9 GHz, 12.5 GHz and 18.5 GHz.

5.4.2 Other planar inductors results

Fig. 5.8 shows the increase of inductance value and Q-factor for all measurable inductors in the meander or spiral topologies. With the theoretical model explained above, we should get a same value of increase of the inductance or Q-factor, while the resistance value is the same. Some samples have higher increase of Q-factor than inductance (M2a, M3a, M1d, M2d, M3d), while other have smaller increase (M4a, M1b, S2a, S3a, S4d). We could understand those differences on the assump-

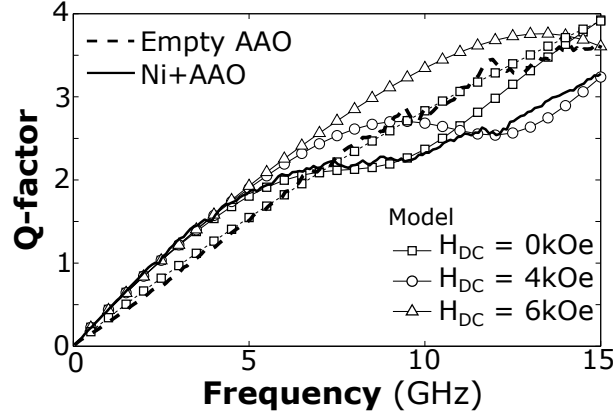


Figure 5.7 – Q-factor of M1c inductor for empty (dashed curves) and nano-wired (solid curves) substrates. Curves with markers are the numerical characterization at different applied magnetic fields.

tion of better or worse contacts of the measurements, respectively. The consequence is a varying resistance value.

5.5 Optimization

Different strategies offer to optimize the Q-factor. In the case of nanowired substrate, we could maximize the presence of the ferromagnetic material by increasing the packing density and the length of the wires. Secondly, an optimized fabrication process could reduce the losses of the AAO membrane. Insertion losses can also be minimized via the increase of the thickness of electrodes, up to the minimal skin depth value over considered frequency range (i.e., $1.45 \mu\text{m}$ at 4 GHz for aluminum with conductivity of $3 \cdot 10^7 \text{ S/m}$). Geometrical optimization of inductor at fixed length could also increase the inductance value without increasing the resistance of the system. It could optimize the quality factor.

As mentioned previously, the fabricated device has nanowires filling in average 40% of alumina membrane, yielding an increase of quality factor of about 23% at 4 GHz. In Fig. 5.9, the increase of the nanowires length from $h_w = 40 \%$ to the ideal case of $h_w = 100 \%$ improves the quality factor enhancement at 4 GHz from 23 % to a value of 35 %.

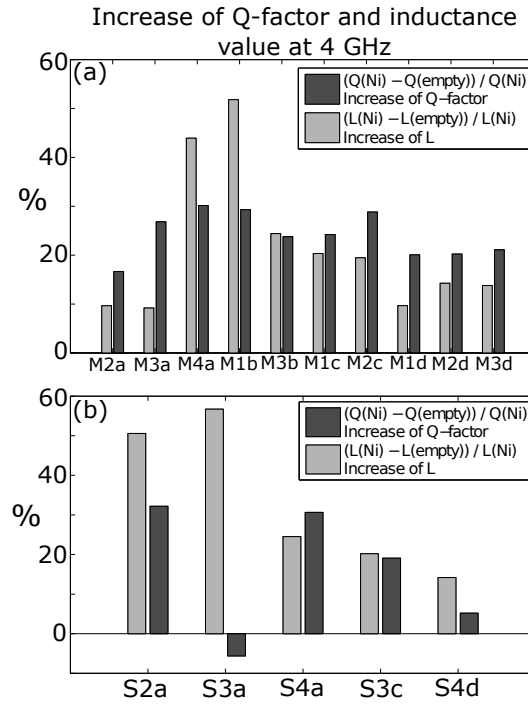


Figure 5.8 – Increase of Q-factor and inductance value at 4 GHz for all the measured (a) meander and (b) spiral inductors.

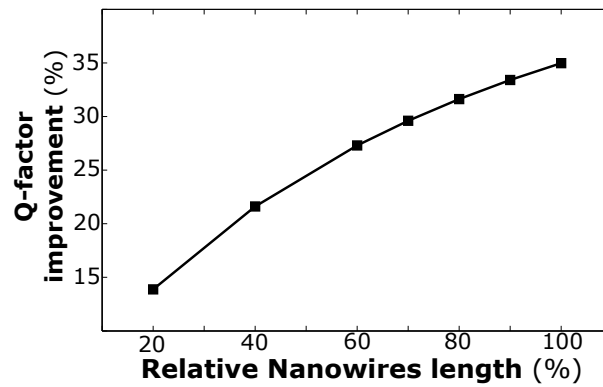


Figure 5.9 – Numerical optimization of the increase of Q-factor with the length of the nanowires.

5.6 Conclusions

Arrays of ferromagnetic nanowires embedded into a self-supported AAO membrane provide a lot of advantages for the conception of planar inductors. We observed a simultaneous increase of the inductance value and the quality factor up to 7 GHz, thanks to the large amount of ferromagnetic material and the nanoscale of the wires limiting conducting losses. Our devices show the largest increase reported in literature for such ferromagnetic inductors: +30 % for the inductance value and +23 % for the quality factor. Increase of the capacitance value with the presence of nanowires in the same proportion of the calculated effective permittivity proves the experimental validation for high porosity arrays of the model [35]. The analytical model is in good agreement with the experimental results, which permits a numerical characterization to improve or to tune the microwave properties of planar inductors. Even more, this technology offers great advantages as an easy integration in miniaturized topologies and its tunability to a magnetic field to broaden the desired frequency range. Our analytical model predicts that the increase of quality factor can be maximized up to 35 % by an optimization of the pores filling.

Chapter 6

Enhanced magnetic anisotropy of Ni nanowire composite arrays

6.1 Introduction

Arrays of parallel magnetic nanowires embedded in non-magnetic dielectric templates are of considerable interest for fundamental physics studies and for their possible exploitations in relevant applications. Compared to other magnetic particle/dielectric composites, nanowire arrays embedded in nanoporous membranes allow controlling the magnetic material, nanowire height and diameter, and packing fraction. Particularly, the packing fraction modulates the magnitude of the dipolar interaction field that plays a key role in the magnetic properties of the assembly. Proper control and tuning of their magnetic properties requires further understanding of the interplay between intrinsic and shape effects as well as the interaction between the nanowires and the host matrix.

Magnetoelastic (ME) effects provide an additional parameter to control the magnetic properties in assemblies of nanowires because of the large surface to volume ratio, thus enhancing the mechanical coupling between the two phases. Although the ME contribution is generally weak at room temperature, it can reach the same order of magnitude as for instance the shape anisotropy at low temperatures in specific systems, as already shown in Ni nanowires embedded in polycarbonate membranes [66, 67, 68]. Besides, recent studies suggest the existence of magnetoelastic effects in Ni nanowire arrays embedded in porous alumina membranes [69, 70]. In addition to these thermal characterizations, boosted magnetic anisotropy has been observed at room temperature in Ni nanowire

arrays embedded in alumina membrane with small diameters of pores. Double FMR absorption peaks indicate the presence of two preferential anisotropies inside the Ni nanowires and the large FMR field shifts show an enhancement of the anisotropy compared to the magnetostatic contribution.

This change in the magnetic anisotropy, induced by a deformation of the Ni structure, does not concern the NiFe and CoFe nanowires since these kind of magnetic materials have negligible magnetostriction and magnetocrystalline coefficients.

6.2 Theoretical prediction and extraction of magnetic anisotropy energies

In this section, we summarize the magnetic anisotropies of Ni nanowire arrays that have been explained in the section 1.1.2. Theoretical formulas allow to predict the value of magnetic energies for such anisotropy. An equation will extract from the FMR measurements the energy of the effective anisotropy existing in such nanowire array. For a typical Ni nanowire array, magnetostatic anisotropy (MS), including the shape anisotropy and the dipolar interaction, is the main contribution at room temperature. Magnetocrystalline (MC) and thermally-induced magnetoelastic (ME^{therm}) contributions may be considered negligible. This assumption changes under cooling the temperature until 77K, while the MC and ME anisotropy values become the same order of magnitude than the MS anisotropy. A specific case of magnetic contributions is also observed for small diameter Ni nanowire arrays embedded in AAO template.

The estimation of the different contributions to the magnetic anisotropy is expected to provide some insights to the behavior observed in FMR measurement. The effective anisotropy energy K_{eff} can be extracted from the FMR measurements using the formula:

$$K_{eff} = \frac{f_{H=0} \cdot M_s}{2\gamma}, \quad (6.1)$$

where $f_{H=0}$ is the zero-field resonance frequency, $M_s = 485 \text{ emu}\cdot\text{cm}^{-3}$ is the saturation magnetization at room temperature and $\gamma = 3.09 \text{ GHz}\cdot\text{kOe}^{-1}$ the gyromagnetic ratio for Ni.

As mentioned in the section 1.1.2 and resumed in table 6.1, energies can be calculated for the MS, the MC and the ME^{therm} contributions.

Extraction of effective energy K_{eff}	
	$K_{eff} = \frac{f_{H=0} \cdot M_s}{2\gamma} \quad (6.2)$
Theoretical predictions	
MS energy	$K_{ms} = \pi M_s (1 - 3P) \quad (6.3)$
MC energy	$K_{mc} = \frac{K_1}{4} \quad (6.4)$
ME ^{therm} energy	$K_{me}^{therm} = \frac{3\lambda_{110}\sigma}{2} \quad (6.5)$
	$\epsilon = \frac{(1 - P) E_{MATRIX}}{(1 - P) E_{MATRIX} + P E_{Ni}} (\alpha_{MATRIX} - \alpha_{Ni}) \Delta T \quad (6.6)$

Table 6.1 – Formula for extracting the effective energy K_{eff} from FMR measurements, calculating the magnetostatic K_{ms} , the magnetocrystalline K_{mc} and the thermal magnetoelastic K_{me}^{therm} energies.

- Magnetostatic anisotropy (MS): K_{ms} is calculated by the formula $K_{ms} = \pi M_s^2 (1 - 3P)$ with M_s the saturation magnetization and P the packing fraction of nanowire array. M_s increases when temperature decreases.
- Magnetocrystalline anisotropy (MC): for the Ni *fcc* nanowires, K_{mc} is calculated by the formula $K_{mc} = K_1/4$. K_1 is temperature-dependent. Values of K_1 is visible in Fig. 1.5. This anisotropy energy is independent on the porosity.
- Thermally-induced magnetoelastic anisotropy (ME^{therm}): the mismatch of the thermal expansion between template (α_{MATRIX}) and nickel (α_{Ni}) under cooling induces a strain on the wire, which modifies the anisotropy energy. K_{me}^{therm} is calculated by eq. (6.5), with

$\sigma = E_{Ni}\epsilon$ and taking into account the temperature dependence of the magnetostriction coefficient (λ_{110}). The strain ϵ can be calculated using eq. (6.6) with E_{MATRIX} and E_{Ni} the Young's modulus for the template and for the Ni.

When the effective energy extracted from FMR measurements does not correspond to the predicted energy including MS, MC and ME contributions, an excess energy can be calculated using $K_{excess} = K_{eff} - K_{ms} - K_{mc} - K_{me}^{therm}$. K_{excess} represents an additional contributions to the magnetic anisotropy, as it will be presented for small diameter Ni nanowire arrays embedded in AAO template.

In table 6.1, temperature-dependent coefficients are observed for several parameters. Values at room temperature (300 K) and liquid nitrogen temperature (77 K) are reported in table 6.2.

	300 K	77 K
M_s [emu/cm ³]	485	517
$K_1 \cdot 10^{-4}$ [erg/cm ³]	-5.2	-84.3
$\lambda_{110} \cdot 10^{-6}$	-34	-54

Table 6.2 – Values of parameters for nickel at room and liquid nitrogen temperatures: saturation magnetization M_s , cubic magnetocrystalline coefficient K_1 , and (110)-direction magnetostriction coefficient λ_{110} .

6.3 Temperature-dependent magnetic anisotropy in Ni nanowire composite arrays

In this section, Ni nanowire arrays embedded into nanoporous membranes with different thermal expansion and elastic properties have been investigated. This section refers to Ni nanowires of relatively large diameter (50 - 200 nm). The contrasting results obtained on the small diameter nanowires will be discussed in the next section. It is shown that the thermally induced magnetoelastic effects strongly depend on the packing fraction as well as on the thermal expansion coefficient mismatch between the host matrix and metal nanowires. In order to account for the variation of the ME^{therm} energy on the parameters mentioned above, a simple model that considers the influence of the nanowires packing fraction and the matrix-assisted deformation process has been proposed. The comparison between the calculated data and the experimental results shows

a fairly good agreement. Thus providing support for this tendency prediction model, which can be used to tailor magnetic and magnetoelectric properties in such nanocomposite materials.

6.3.1 Samples used in the present study

Arrays of Ni nanowires have been grown by electrodeposition into the pores of selected nanoporous membranes. The host polymer matrix consists in 25 μm thick track-etched membranes made of PC and PVDF with diameter φ in the range 50 nm - 200 nm. Samples with different packing densities P between 1% and 12% were fabricated in order to examine the influence of wire density on the magnetoelastic anisotropy energy. Additional 50 μm thick AAO membranes ($\varphi = 21, 100$ nm and $P = 12, 15\%$) was also used as a template.

6.3.2 FMR measurements at different temperatures

Fig. 6.1(a) shows FMR absorption spectra recorded at 30 GHz at different temperatures for an array of Ni nanowires embedded in a PVDF membrane with $\varphi = 105$ nm and $P = 1.7\%$. It can be seen that the shift in the resonance field $\delta H_r = H_r - H_{r(295\text{K})}$ where H_r is the resonance field at the temperature T and $H_{r(295\text{K})}$ its value at 295 K is negative by cooling below room temperature ($\delta H_r \sim -6.5$ kOe at 77 K) and positive with increasing the temperature ($\delta H_r \sim +0.85$ kOe at 345 K). Such behaviors are consistent with the presence of significant ME^{therm} effects as the temperature is modified, which is due to the Ni and the polymer thermal expansion coefficient mismatch [67, 68].

In contrast, a much weaker temperature dependent FMR was measured on the Ni-AAO sample, as concluded by the small positive shift of the resonance field ($\delta H_r \sim 0.2$ kOe) at 77 K (Fig. 6.1(a)-dotted lines) thus indicating a reduction of the total anisotropy along the wire axis. In the case of Ni-AAO, other energetic contributions ensure the positive shift.

Since the thermal expansion coefficient for Ni is lower than the one for the polymer matrix, their mismatch induces a resultant stress under cooling that is exerted by the polymer matrix on the Ni nanowires. As a result, the Ni nanowires tend to contract or to expand less than the polymer matrix under cooling or under heating, respectively. Besides, for an array of metallic nanowires with a low packing fraction embedded in a polymer matrix, the axial stress is much larger than the radial stress [66], then such temperature variations lead to a significant compressive stress exerted by the polymer membrane along the nanowires axis upon cooling

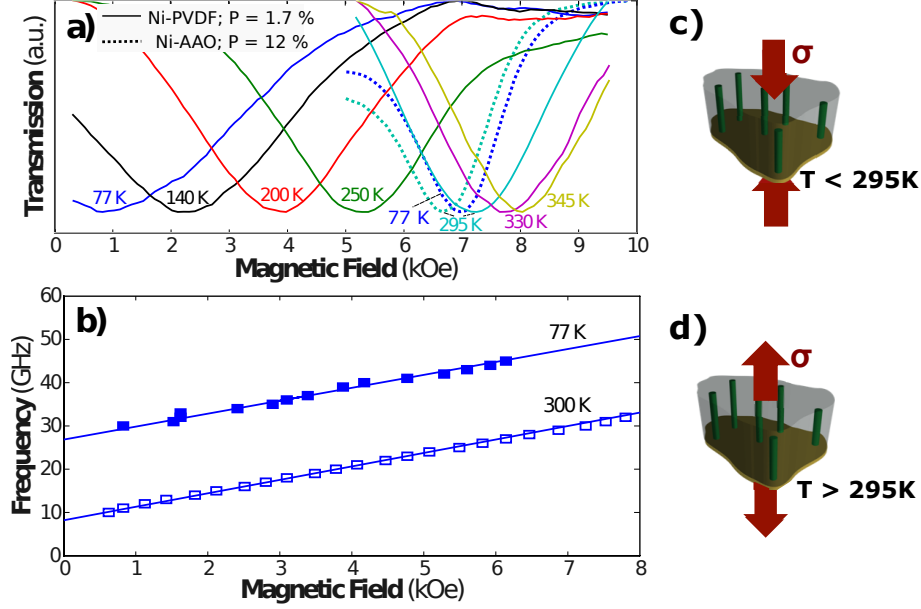


Figure 6.1 – (a) FMR absorption spectra measured at 30 GHz as a function of temperature for Ni nanowires embedded in a PVDF membrane with $P = 1.7\%$ (solid lines) and embedded in an AAO template with $P = 12\%$ (dotted lines); (b) dispersion relations for the Ni-PVDF sample measured at 77 K (filled symbols) and 300 K (open symbols). (c) compressive and (d) tensile stress on the Ni nanowires (green) induced by the polymer membrane in the case of cooling or heating the sample respectively.

and to a tensile stress upon heating. Interestingly, we observed that in these measurements δH_r does not depend on the excitation frequency. As illustrated in the Fig. 6.1(b), the dispersion relations measured at 295 K and 77 K for the Ni-PVDF sample are parallel to each other, which leads to a full determination of the magnetoelastic energy K_{me}^{therm} from absorption spectra measured at a given constant frequency.

6.3.3 Model and interpretation of experimental results

From previous works, it is known that the Ni nanowires fabricated following this procedure have a preferred orientation along the [110] direction, which is along the wires axis [66, 113, 114]. Therefore, at low temperature, the effect of the first MC anisotropy constant becomes large enough to contribute significantly to the total anisotropy energy. Moreover, above a certain diameter of the wire (~ 50 nm), it was shown that no significant change in the ME^{therm} effects occur with the variations

in Ni wire diameter while P is kept constant [67], so that packing density and membrane material are the more relevant parameters. Using the same approach as in a previous study [67], we may express K_{me}^{therm} , using the resonance field obtained from FMR measurement at room and low temperatures and including the theoretical predictions of MS and MC anisotropy contributions, as follows:

$$K_{me} = \frac{M_s}{12} \left[-A + \sqrt{A^2 - 8 \left(\frac{\lambda_{100}}{\lambda_{111}} + 1 \right) (B - B_{295K})} \right]. \quad (6.7)$$

In this equation, $A = (H_r + H_{ms} - 2H_{A1}) (\lambda_{100}/\lambda_{111}) + 3H_r + 3H_{ms}$, $B = (H_r + H_{ms})^2 - H_{A1} (H_r + H_{ms} + 2H_{A1})$; $H_{ms} = 2\pi M_s (1 - 3P)$ is the MS field including shape and dipolar interactions terms [23], M_s is the saturation magnetization, $H_{A1} = K_1/M_s$, K_1 is the first order anisotropy constant, λ_{100} and λ_{111} are the negative magnetostriction coefficients in the directions [100] and [111] for Ni and B_{295K} is the value of B at room temperature. Using equation (6.7), the variation of K_{me}^{therm} with T was deduced from FMR resonance fields using known values for bulk Ni of M_s [115], K_1 [77], λ_{100} and λ_{111} [78]. Since the nanowire diameter considered here ranges from 50 to 200 nm, the values of the magnetic parameters correspond to those of the bulk. The temperature evolution of K_{me}^{therm} obtained from the FMR measurements done on the Ni nanowire arrays grown in PVDF and PC membranes with selected packing densities (around 2% and 5-6%) is reported in Fig. 6.2.

It can be observed that K_{me}^{therm} increases monotonically with decreasing temperature in all cases. From Fig. 6.2 it also appears that K_{me}^{therm} is enhanced for Ni-PVDF samples (squares) in comparison to Ni-PC samples (circles) considering similar P values. Besides this, for the two sets of Ni nanowire samples embedded in polymer host membranes, the magnitude of K_{me}^{therm} decreases as P is increased for temperature values below room temperature, in good agreement with previous results [67].

The enhanced ME^{therm} effect in the PVDF based samples can be qualitatively understood by the larger mismatch of thermal expansion coefficients between the Ni nanowires ($\alpha_{Ni} = 1.3 \cdot 10^{-5} \text{ K}^{-1}$) and PVDF membrane ($\alpha_{PVDF} = 12 \cdot 10^{-5} \text{ K}^{-1}$) than between the metal nanowires and PC membrane ($\alpha_{PC} = 7 \cdot 10^{-5} \text{ K}^{-1}$). For the Ni-PVDF sample with $P = 1.7\%$, K_{me} at 77 K is significantly large ($\sim 11.2 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$), and is therefore found to dominate over the evaluated MC energy in the [110] direction [76], $K_{mc} = K_1/4 \sim -2 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$ and the MS energy $K_{ms} = \pi M_s^2 (1 - 3P) \sim 7.8 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$ at 77 K. In Fig. 6.3(a)-blue squares, $K_{excess} = 10 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$ at 77 K, which represents the excessive energy at low temperature compared to the room temperature,

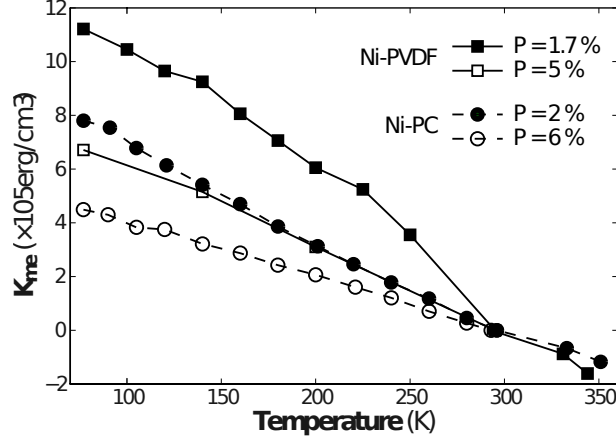


Figure 6.2 – Variation of K_{me} with T for Ni nanowire arrays with different packing densities embedded in PC and PVDF templates. Ni-PVDF with $P = 1.7\%$ (filled squares) and $P = 5\%$ (open squares); Ni-PC with $P = 2\%$ (filled circles) and $P = 6\%$ (open circles).

is similar to the K_{me}^{therm} . At room temperature, $K_{ms} \sim 7 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$ while both K_{mc} and K_{me}^{therm} are negligible, then the large increase in the uniaxial anisotropy at low temperature is primarily mediated by the ME^{therm} anisotropy. It is interesting to note that when heated above room temperature, both PVDF and PC based Ni nanowire samples display a negative ME^{therm} energy. In that case the wires are forced to expand together with the polymer matrix, thus resulting in a tensile stress along the wire axis. A moderate increase of 50°C in the sample temperature can significantly shift the FMR field by about 1 kOe (Fig. 6.1(a)), which in turn leads to a decrease of K_{me}^{therm} , as shown in Fig. 6.2. The ME^{therm} energy strongly depends not only on the temperature but also on both the packing density and the membrane material. Therefore, all these quantities can be considered together as input parameters in a simplified model for the ME^{therm} energy.

Fig. 6.3 compares the contributions to the magnetic anisotropy as a function of the temperature for (a) Ni nanowire array inside a PVDF membrane with porosity $P = 1.7\%$ and for (b) Ni nanowire array in an AAO membrane with porosity $P = 12\%$. The excess energy K_{excess} originates from the addition of three temperature dependent energy terms,

$$K_{excess} = K_{ms} + K_{mc} + K_{me}^{therm}, \quad (6.8)$$

namely the MS energy $K_{ms} = K_{ms}(T) - K_{ms}(300\text{K})$, the MC anisotropy energy K_{mc} and the ME^{therm} anisotropy energy K_{me}^{therm} . In the case of

the low-density Ni-PVDF system, the main contribution to the excess energy is provided by the huge ME^{therm} anisotropy due to the large mismatch in the thermal expansion coefficients between Ni and PVDF.

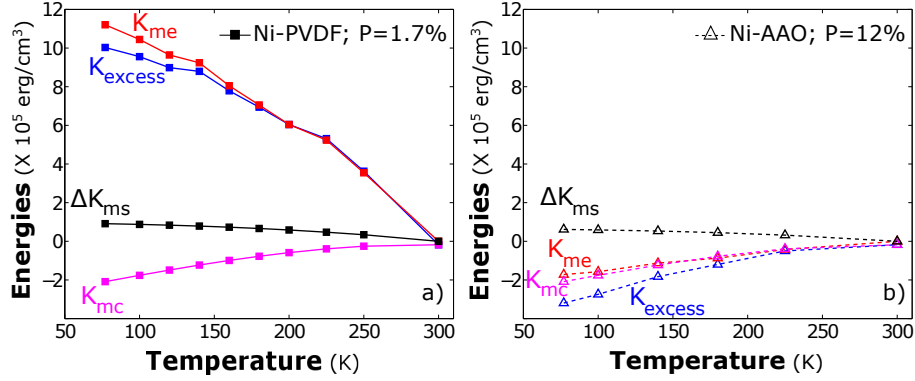


Figure 6.3 – Energy contributions for (a) PVDF-Ni and (b) AAO-Ni samples for different temperatures. Excess energy K_{excess} is the sum of all additional contributions compared to the case at room temperature: the MS energy $\Delta K_{ms} = K_{ms}(T) - K_{ms}(300K)$, the MC anisotropy energy K_{mc} and the ME^{therm} anisotropy energy K_{me}^{therm} .

In contrast, magnetoelasticity is not the main contribution to the magnetic anisotropy for arrays of Ni nanowires embedded in AAO membranes of porosity $P = 12\%$. At 77 K, the MC energy ($K_{mc} = -2.05 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$) has a similar value to the ME^{therm} energy ($K_{me}^{therm} = -1.95 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$). Small change in MS energy ($\Delta K_{ms} = 0.5 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$) is observed between 77 K and room temperature. Therefore, $K_{excess} = -3.2 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$ at 77 K for Ni-AAO with $P = 12\%$.

An estimation of the thermally-induced magnetoelastic anisotropy K_{me}^{therm} can be made through [116, 117]

$$K_{me}^{therm} = \frac{3\lambda_{110}\sigma}{2}, \quad (6.9)$$

where $\sigma = E_{Ni}\epsilon$ is the axial stress induced on the Ni nanowires and ϵ is the strain, $E_{Ni} \sim 214 \text{ GPa}$ is the Young's modulus for Ni, $\lambda_{110} \sim -34 \cdot 10^{-6} \text{ K}^{-1}$ is the room temperature saturation magnetostriction constant ($\lambda_{110} \sim -54 \cdot 10^{-6} \text{ K}^{-1}$ at 77 K). To calculate the dependence of the matrix-assisted strain on the packing fraction for arrays of parallel Ni nanowires embedded in a host matrix, we used the following expression

[117]:

$$\epsilon = \frac{(1 - P) E_{\text{MATRIX}}}{(1 - P) E_{\text{MATRIX}} + P E_{\text{Ni}}} (\alpha_{\text{MATRIX}} - \alpha_{\text{Ni}}) \Delta T, \quad (6.10)$$

where ΔT is the change in temperature, α_{Ni} and α_{MATRIX} are the thermal expansion coefficients for Ni and the host matrix respectively (with $\alpha_{\text{AAO}} \sim 10^{-5} \text{ K}^{-1}$ the estimated thermal expansion coefficient for amorphous alumina [118]) and E_{MATRIX} is the Young's modulus for the host matrix ($E_{\text{PC}} \sim E_{\text{PVDF}} \sim 2.3 \text{ GPa}$; $E_{\text{AAO}} \sim 350 \text{ GPa}$). As a rough estimate, we will use α -values at room temperature to calculate ϵ . For the Ni-PVDF sample with $P = 1.7\%$, the calculated strain $\epsilon \sim 0.9\%$ in the direction of the wire axis when the temperature varies from 295 K to 77 K, is equivalent to an axial compressive stress $\sigma \sim 1.9 \text{ GPa}$. Fig. 6.4 shows the variation of the calculated K_{me}^{therm} at 77 K as a function of packing density P considering the different host nanoporous templates. The estimated variation is compared to the experimental values extracted from FMR measurements using Eq. (6.7), where a very good agreement is found. Error bars for the packing fraction arise from the uncertainty generated by the pores size distribution, as determined by SEM observations over large areas. Not only large positive values of K_{me}^{therm} are obtained for low-density Ni nanowire arrays grown in polymer templates but also an increase in packing density causes a rapid drop in K_{me}^{therm} . Such features can be understood in terms of the matrix-assisted compressive stress on the Ni wires upon cooling and to the P dependence of the strain reflected by the much lower elastic modulus in polymers than in metals. Therefore, the magnitude of the magnetoelastic anisotropy energy depends strongly on the effective volume ratio of the polymer to the metal nanowire. The enhancement of K_{me}^{therm} for the Ni-PVDF samples in comparison to Ni-PC samples with similar P values by about a factor 2 is due to the fact that the former has a larger thermal expansion coefficients mismatch than the later. In contrast, for the Ni-AAO sample with $P = 15\%$, the small shift in the absorption peak to higher field values at 77 K indicates a higher negative value of K_{me}^{therm} since the thermal expansion coefficient for amorphous alumina is slightly smaller than that for nickel. Therefore, a relatively low tensile stress on Ni nanowires occurs upon cooling from room temperature. In addition, the relatively small difference in Young's modulus between Ni and alumina leads to a much weaker P dependence of K_{me}^{therm} . Using the above-mentioned values, one can estimate $K_{me}^{\text{therm}} \sim -1.4 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$ at 77 K, which has to be compared to $K_{mc} \sim -2 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$ and to the magnetostatic energy increases $\delta H_{ms} = K_{ms}(77\text{K}) - K_{ms}(295\text{K}) \sim 0.5 \cdot 10^5 \text{ erg}\cdot\text{cm}^{-3}$. Thus overall, a reduction of the effective anisotropy along the wire axis

occurs gradually upon cooling.

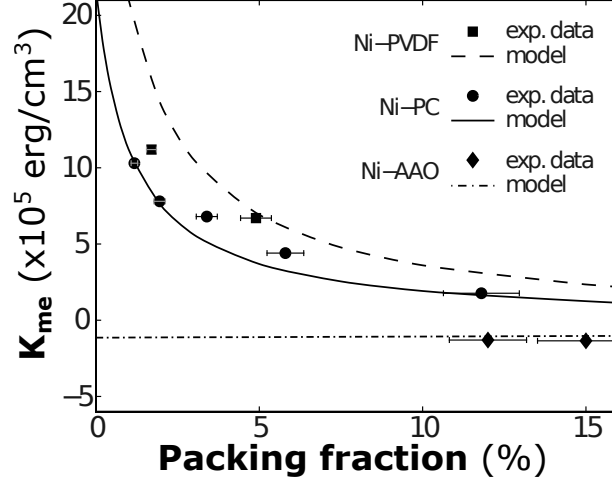


Figure 6.4 – Experimental and calculated thermally-induced magnetoelastic anisotropy energy K_{me}^{therm} as a function of the packing density for Ni-PVDF, Ni-PC and Ni-AAO samples.

6.3.4 Conclusions

In arrays of Ni nanowires electrodeposited into porous PVDF and PC membranes, large ME^{therm} effects have been observed as the temperature is modified from room temperature, which are attributed to the thermal expansion coefficients mismatch between the Ni nanowires and the membrane. Particularly, the ME^{therm} energy is larger for the Ni-PVDF than for the Ni-PC nanocomposites as a result of the larger thermal expansion coefficients mismatch of the former. On the contrary, lower ME^{therm} effects have been observed in Ni nanowires grown into nanoporous alumina membranes, which is ascribed to the relatively lower thermal expansion coefficients mismatch between the nanowires and the alumina membrane. In order to account for these effects, a simple model for the ME^{therm} anisotropy energy that depends on the parameters like the packing density, the coefficients of thermal expansion and elastic modulus of constituents, and the temperature has been proposed and a very good agreement with the experimental data has been obtained. This demonstrates that the magnetic anisotropy of arrays of ferromagnetic nanowires embedded in nanoporous membranes is tunable by an appropriate choice of the parameters mentioned above, which is of considerable importance for the design of magnetically adjustable devices based on nanocomposites.

6.4 Diameter-dependent magnetic anisotropy in Ni nanowire composite arrays

As previously observed in our research group at UCL [22], enhanced anisotropy has been measured in arrays of small diameter Ni nanowires embedded in AAO membranes by the observation of large shifts of resonance peaks to low field compared to the theoretical predictions calculated considering only the magnetostatic field $H_{ms} = 2\pi M_s (1 - 3P)$. An additional magnetic anisotropy induces these changes when considering small diameter Ni nanowires. This unexplained effect had been observed in several small diameter Ni nanowires embedded in AAO membranes. In his previous work [22], J. De La Torre Medina already suggested that magnetoelastic effects could be present at room temperature for this type of nanowired samples. The deformation of the lattice may be induced during the electrodeposition process and both strained and unstrained crystallographic structures may coexist in such long Ni nanowires.

In this work we report on new results obtained on small diameter Ni nanowire arrays and we propose a more detailed model to explain this enhanced anisotropy. We suggest that the electrodeposition process in extrinsically confined media ($\varphi < 50$ nm) leads to some deformation in the Ni nanowire structure. This effect is more pronounced when the thin nanowires display highly oriented (110) texture along wire axis. This deformation may result from the mismatch in the lattice coefficients between Ni nanowires and the alumina membrane as well as from the stiffness of the alumina pore walls.

In our hypothesis, Ni nanowires could be approximated as a two-stages system with polycrystalline and single crystal phases in series, as already demonstrated for Sb nanowires in [119] and for Ni nanowires in [120, 121]. Fig. 6.5(a-d) shows the TEM image of two parallel Sb nanowires with the lattice fringes derived from the crystalline grains for different positions on the nanowires (Fig. 6.5(b-d)). Au material represents the cathode of the AAO membrane and therefore the electrodeposition process starts at this point. Due to the electrodeposition process, the nucleation of the wires at the bottom of the pores is a 3D growth transforming the first stage of the wire as a polycrystalline material (Fig. 6.5(b) - (1)). Nanowires growth shows a transition area after a certain length, which is symbolized by the apparition of a single crystal grain over all the wire section (Fig. 6.5(c) - (2)). After this length, nanowires shows clearly 2D lattice fringes indicating the single-crystalline characteristic of the wire (Fig. 6.5(d) - (3)). The process of the growth from the bottom of the pores is represented in the sketch of Fig. 6.5(e) where

polycrystalline grains start growing on the cathode and the growth of single crystal along a preferential plane comes after a certain length. In our hypothesis (Fig. 6.5(f)), the first stage is polycrystalline, low ordered atomic structure with a small (or zero) deformation, while the second stage is highly ordered in the (110) crystallographic direction along the wire axis. A deformation induced by the lattice coefficients mismatch between Ni and alumina may exist in all the wire section in this second stage.

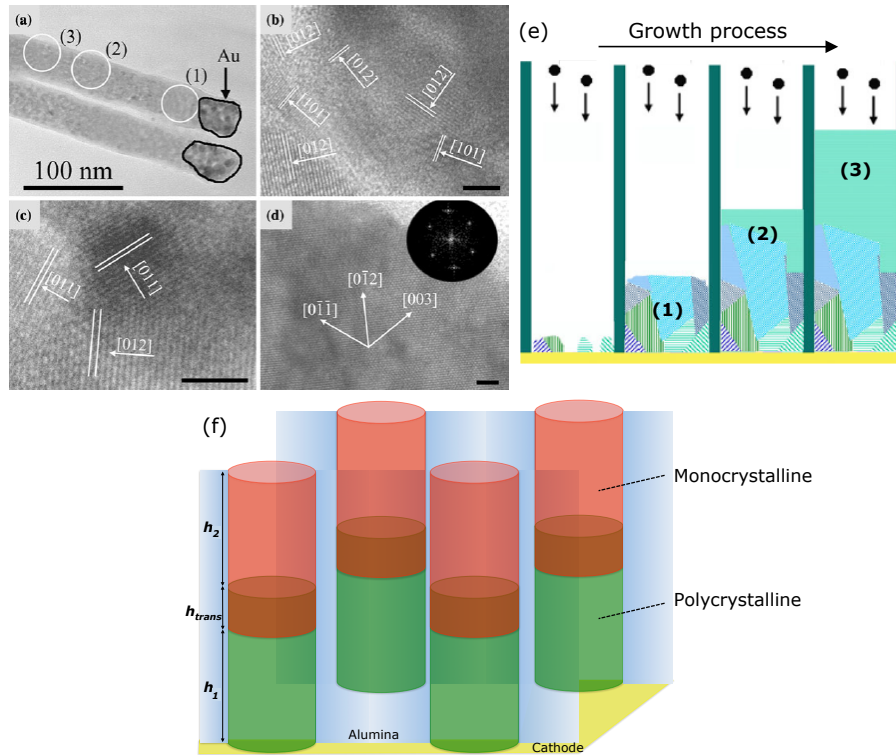


Figure 6.5 – (a)-(d) TEM image of two parallel Sb nanowires with Au cathode at the beginning. (b), (c) and (d) are HRTEM images of the areas marked (1), (2) and (3) respectively. The inset in (d) is its FFT image. (e) Sketch of the nucleation and growth process of the metal nanowire grown by pulsed electrodeposition where the three areas marked in (a) can be observed. (f) Sketch of our hypothesis with two-stages system for Ni nanowires. The first stage (green part) is not deformed, while the second stage (red part) is deformed. h_1 and h_2 represent the length of the first and second stage, respectively. The experimental results indicate that the second stage of the nanowires is compressed. (a)-(e) from [119].

The two-phases hypothesis allows to explain the existence of the double peaks structure in the FMR absorption curves observed for several small diameter nanowire arrays. It also explains the dependence of relative intensities of the two absorption peaks on the length of the nanowires in the 21 nm-diameter Ni-AAO samples. Dependence to the nanowires length has already been observed in [122] through the aspect ratio of Ni nanowires. Fig. 6.6 shows the dependence of the coercive field obtained by XRD measurements on the aspect ratio of 25 nm-diameter Ni nanowires for two densities of wires. The increase of the coercive field demonstrates the enhancement of the magnetic anisotropy.

A similar growth process could be seen in the case of NiFe and CoFe nanowires where two stages (poly- and mono-crystalline) appear along the wire. This particularity is covered up in the FMR measurements or in the hysteresis loops by the negligible values of the magnetostriction and magnetocrystalline coefficients of the NiFe and CoFe alloys used in this work. XRD measurements should be done to confirm the higher crystallinity of small diameter NiFe or CoFe nanowires.

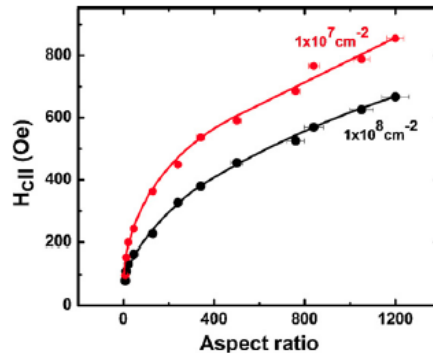


Figure 6.6 – Coercivity values of Ni nanowires as a function of fluence of nanowires, when the field was applied parallel to the wire axis. From [122].

6.4.1 Room temperature experimental results and energy considerations

The characterization of Ni nanowires in AAO membranes with diameter in the range 18 nm and 100 nm shows the different magnetic anisotropies values through FMR measurement for the different samples. X-ray diffraction (XRD) and magnetometer measurements complete the comparison between large and small diameter Ni nanowire arrays.

Anisotropy at room temperature

Fig. 6.7 compares the absorption spectra for Ni nanowire arrays embedded in AAO membranes with different diameters. Packing fractions are in the range 10 to 15%. The feature of absorption curves for the Ni nanowire arrays embedded in AAO membrane is interesting since it shows a shift to lower resonance field for smaller diameters and the apparition of a double peak structure. The absorption peaks represent the distribution of resonance fields, which in turns are associated to the distribution of anisotropy fields. Therefore, absorption spectra with double peak structure indicate that the nanowires in the array present several magnetic anisotropies. Such double peaks were already reported in the case of a non-saturated array where nanowires have up or down magnetization [28] and in the case of Co nanowires exhibiting inhomogeneous magnetocrystalline anisotropy contributions [26].

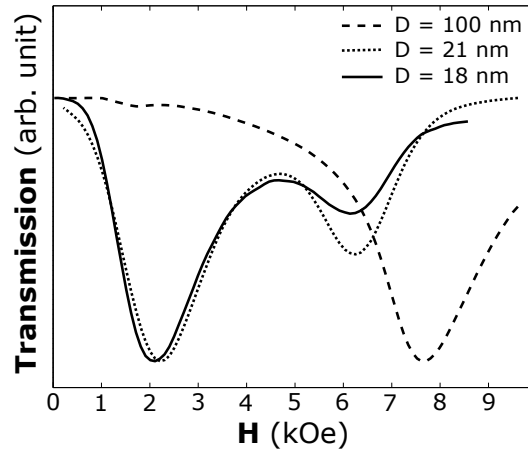


Figure 6.7 – Absorption spectra at room temperature for Ni nanowire arrays in nanoporous alumina membranes at 28 GHz with similar porosities : 100 nm and 15% (dashed line), 21 nm and 12% (dotted line) and 18 nm and 10% (solid line).

As shown in Fig. 6.7, a shift of 5.5 kOe appears between the main absorption peak of the 100 nm-diameter sample and the one of the 18 nm-diameter sample. Similar large shifts in the resonance field have already been shown at low temperature for low-density Ni nanowire arrays embedded in polymer membranes when magnetoelastic anisotropy was induced by the large mismatch in the thermal expansion coefficients between Ni nanowires and the template. As suggested hereafter, the enhanced anisotropy for small diameter Ni nanowires embedded in AAO templates might also be a room temperature magnetoelastic contribu-

tion (ME^{RT}) (Fig. 6.5). Ni nanowires grown by electrodeposition are known to have the preferential crystallographic orientation $[110]$ parallel to the wire axis [66]. At room temperature, the magnetostriction of the $[110]$ is negative $\lambda_{110} = -34 \cdot 10^{-6}$. Therefore, an enhancement of the anisotropy, as observed in small diameter samples, would be associated to a compression of the Ni nanowires.

It is worth mentioning the particular case of the two 21 nm-diameter samples that shows two different absorption spectra. The only difference consists in the length of the nanowires, which is roughly 1.3 times longer for sample (1) than for sample (2) (Fig. 6.8). Both samples exhibit a double absorption peak, but the relative intensity of the peaks differs in the two samples. Considering the absorption peaks as the contribution of the two stages existing in a confined nanowire (Fig. 6.5(f)), relative intensity of the low resonance field peak becomes larger with the increase of nanowires length. This special feature of both samples seems to be a change in the main preferential anisotropy contribution. As shown in the dispersion relation (Fig. 6.8(b)), both absorption peaks evolve with the same dependence.

Considering the above assumption, the high H_r peak is associated to the weakly deformed region located at the bottom of the wires, while the low H_r peak is associated to the highly strained region in the upper part of the nanowires. The length of the first stage might be constant for similar diameter samples. The ratio of the intensities between the low H_r and high H_r peaks is modified as the length of nanowires is changed, in agreement with the volume ratio between the two phases.

XRD characterizations of Ni nanowire arrays

The X-ray diffraction is a non-destructive technique that allows to determine the crystallographic structures of materials thanks to angular spectra of the intensity. Fig. 6.9 shows the intensity of crystallographic directions in angular spectra for Ni nanowire arrays with different diameters (18, 21, 35, 100 nm).

The main peak (220) indicates the preferential grown axis during the electrodeposition compared to other crystallographic directions (111) and (200). We see in the Fig. 6.9 that the amplitude of the diffraction peak increases when the diameter of nanowires decreases from 100 to 18 nm. The ratio between the peak areas (220)/(111) increases from 1.6 for the 100 nm-diameter sample to 35 for the 18 nm-diameter sample. These results demonstrate the highly ordered crystallographic structure with preferentially grown direction (220) for the small diameter samples.

The two-stages system is not visible in the XRD measurements due

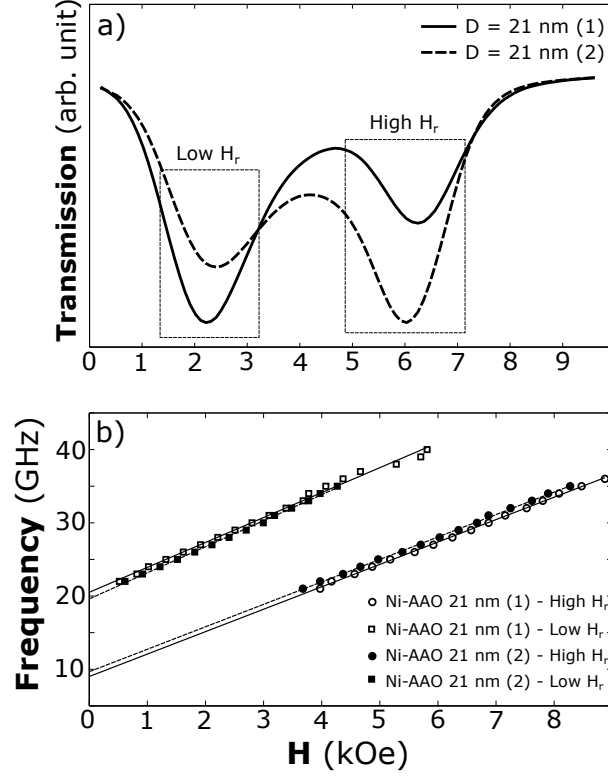


Figure 6.8 – (a) Absorption spectra at room temperature for a 21 nm-diameter Ni nanowire array for two samples at 28 GHz. Porosity of the nanowire arrays is $P = 12$ %. (b) Dispersion relation of both 21 nm-diameter Ni nanowire arrays including both absorption peaks observed in (a).

to the superposition of the two phases. The highly crystallographic and deformed phase would normally show a shift of a few degrees in the peak of crystallographic axis, as observed in [66].

Alternating gradient measurements on Ni nanowire arrays

Additional magnetic characterizations on Ni nanowires using alternating gradient magnetometer were performed. Fig. 6.10 shows the hysteresis loops for a magnetic field H parallel to the nanowires axis of Ni nanowire arrays embedded in a nanoporous alumina template with different nanowires diameters ($\varphi = 18$ and 100 nm).

Wider hysteresis loop and higher remanence values appear when the nanowires diameters decrease. This provides information about the enhanced average anisotropy existing in the small diameter nanowire ar-

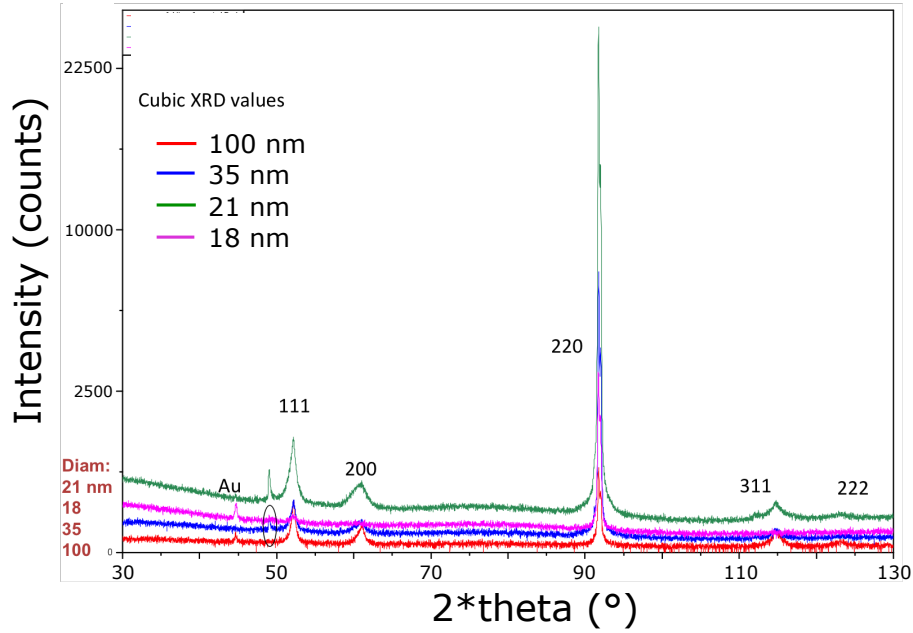


Figure 6.9 – Intensity of crystallographic axis in angular spectra measured by a XRD technique for different nanowires diameters samples: $\varphi = 18, 21, 35$ and 100 nm.

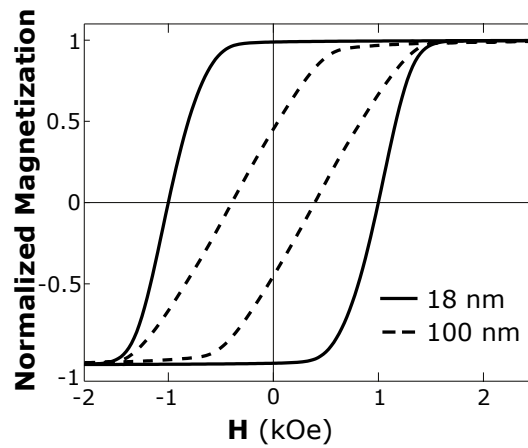


Figure 6.10 – Hysteresis loops for Ni nanowire arrays embedded in nanoporous alumina templates with different pores diameters ($\varphi = 18$ and 100 nm). Packing factors of the nanowire arrays are $P = 10\%$ in the case of 18 nm, and $P = 15\%$ for 100 nm.

rays. Due to the high aspect ratio, nanowires magnetization can only take a bi-stable up-down configuration. The two-phases structures, observed as the double absorption peak in the FMR measurements for 18 nm-diameter sample, does not appear in the hysteresis loop. An high interaction coupling between the two phases should cause the single magnetic field reversal for all the nanowire magnetization, while the dynamic excitation from the rf signal in the FMR measurements could probe the two phases, independently.

Energy considerations of magnetic anisotropy

In the case of large diameter Ni nanowire arrays, MS anisotropy is the main contribution to the effective energy at room temperature, since the MC anisotropy is negligible and ME^{therm} anisotropy is equivalent to zero. The resonance field can be calculated taking into account the relation $H_{ms} = 2\pi M_s(1 - 3P)$, issued from the K_{ms} formula. This formula cannot be used in the case of small diameter Ni nanowire arrays where shifts of the absorption to low field are observed (Fig. 6.7). A characterization of the energies can be done to calculate the value of the excessive energy in those small diameter nanowire samples and to evaluate the compressive strain existing in the two-stages system.

Taking account that this excessive anisotropy energy is caused by a compression on the nanowires at room temperature $K_{excess} = K_{me}^{RT}$ and the assumption that this compression is induced in the axial direction of the wire, we should be able to calculate the strain of those compressed nanowires using the equation

$$K_{me}^{RT} = \frac{3\lambda_{110}\sigma}{2}, \quad (6.11)$$

with $\sigma = E_{Ni}\epsilon$, $E_{Ni} = 214$ GPa, and $\lambda_{110} = (\lambda_{100} + \lambda_{111})/2$, λ_{100} and λ_{111} are the magnetostriction coefficients of the (100) and (111) crystallographic directions respectively.

Table 6.4 reports all the energies that allows to calculate the excessive energy K_{excess} for the Ni nanowire samples with diameter 18 nm, 21 nm and 100 nm. Two values can be written for samples for which a double peak structure has been observed in the absorption (18 and 21 nm).

As we can observe in table 6.4, the excess energy K_{excess} is small for the 100 nm-diameter Ni nanowire array compared to the magnetostatic energy. A negligible magnetoelastic contribution is induced on this kind of nanowire. In the case of small diameters ($\varphi < 50$ nm), the excess energy is twice higher than the magnetostatic energy for the smallest diameters Ni nanowires.

Suggested hypothesis : room temperature magnetoelasticity	
Stress	$\sigma = \frac{2K_{me}^{RT}}{3\lambda_{110}} \quad (6.12)$
Strain	$\epsilon = \frac{\sigma}{E_{Ni}} \quad (6.13)$

Table 6.3 – Suggested hypothesis for the additional contribution.

		$\varphi=18 \text{ nm}$ $P = 10 \text{ \%}$	$\varphi=21 \text{ nm}$ $P = 12 \text{ \%}$	$\varphi=100 \text{ nm}$ $P = 15 \text{ \%}$
H_{res} (Oe)	Low H_r	2018	2213	/
	High H_r	6246	6249	7710
$f_{res,H=0}$ (GHz)	Low H_r	21.1	20.5	/
	High H_r	10.1	9	5.8
K_{eff} [$10^5 \text{erg} \cdot \text{cm}^{-3}$]	Low H_r	16.5	16.1	/
	High H_r	7.9	7	4.6
K_{ms} [$10^5 \text{erg} \cdot \text{cm}^{-3}$]	Low H_r	5.2	4.7	4.1
	High H_r			
K_{mc} [$10^5 \text{erg} \cdot \text{cm}^{-3}$]	Low H_r	-0.1		
	High H_r			
K_{excess} [$10^5 \text{erg} \cdot \text{cm}^{-3}$]	low H_r	11.4	11.5	/
	High H_r	2.90	2.4	0.6

Table 6.4 – Resonance conditions and characterization of energy from anisotropy contributions at room temperature for Ni nanowires in AAO membranes with various pores diameters.

Let's assume that the excess energy is the consequence of magnetoelastic anisotropy in the Ni nanowires at room temperature K_{me}^{RT} . Taking the formula (6.12) and (6.13) giving in table 6.3, the stress σ and the strain ϵ in the axial direction of the wire can be calculated. The results

are given in table 6.5.

		$\varphi=18$ nm	$\varphi=21$ nm	$\varphi=100$ nm
ϵ	Second stage	-1 %	-1 %	/
[%]	First stage	-0.3 %	-0.2 %	negligible data

Table 6.5 – In the case where $K_{excess} = K_{me}^{RT}$.

The data indicate that a compressive stress takes place in the second stage of the small diameter nanowires, corresponding to a strain of about 1 % for the 18 nm and 21 nm sample. This corresponds to the typical value of strain measured at low temperature on a Ni-PVDF sample with $P = 1.6$ % ($\epsilon = 0.9$ % at 77 K). The non-zero values of the strain on the high resonance absorption peaks corresponding to the first stage contribution for 18 and 21 nm-diameters samples could be explained by a strain induced by a small crystallinity of the first stage (Fig. 6.11). An average value of strain can be found for both phases systems thanks to the energetic consideration: $\epsilon_2 = 1$ % and $\epsilon_1 = 0.2$ -0.3 %. The crystallinity in the 100 nm-diameter Ni nanowires is quite low, which prevent deformation of the Ni lattice. No strain is observed in this large diameter nanowires.

6.4.2 Low temperature experimental results

A thermal characterization of one of the 21 nm-diameter Ni nanowire array (Fig. 6.8-dashed line), for which two FMR absorption peaks are observed, records a different behavior in temperature for both absorption peaks. Fig. 6.12 shows the absorption spectra of the 21 nm-diameter samples from liquid nitrogen temperature (77 K) up to the room temperature (300 K) at 30 GHz. Under cooling, the two absorption peaks evolve in different directions: the shifts of the resonance field ΔH_r between room and liquid nitrogen temperature are +300 Oe in the case of the high resonance field, and -1000 Oe for the low resonance field. Opposite shifts for both resonance fields under cooling reveal a difference behavior of preferential anisotropy inducing each FMR absorption. In the case of the low resonance peak (left peak) originated from the compressed second phase, ME^{RT} anisotropy, through thermal dependence of the magnetostriction coefficient (see Fig. 1.6), is the main contribution, while in the case of the high resonance peak (right peak) induced by the first stage, its effective energy at low temperature is the addition of the

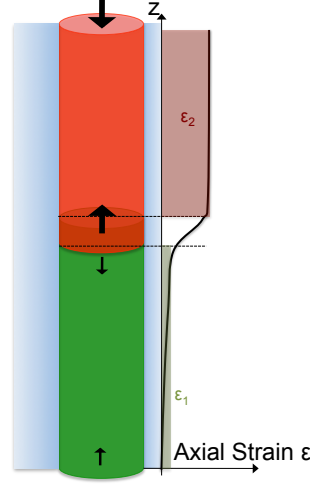


Figure 6.11 – sketch of the two-stages system of a small diameter Ni nanowires embedded in alumina membrane with the schematic of the induced strain ϵ of both systems. An average strain can be calculated for both parts.

MC, MS and ME^{therm} anisotropies contributions.

These results are similar to those obtained on the other 21 nm-diameter sample seen in Fig. 6.8-solid line.

A similar characterization of energies can be done for these results at 77 K. Temperature-dependent coefficients (in table 6.2) have to be adapted to extract K_{eff} and to calculate the MS, MC and ME^{therm} anisotropy energies. At low temperature, the MC anisotropy becomes significant due to the increase of K_1 and should be considered in the calculations. Table 6.6 gives the resonance conditions and energetic characterizations at 77 K for the 21nm-diameter Ni nanowire sample shown in Fig. 6.12.

As previously assumed, strains in the two-stages configuration of Ni nanowires can be calculated thanks to the formula in table 6.3 with the low temperature magnetostriction coefficient (table 6.2). The results are given in table 6.7.

The opposite shifts observed for both absorption peaks upon cooling are then the consequence of a difference in the contributions proportion: for the second stage, the large ME^{RT} contribution induces a negative shift upon cooling, because of the increase of the magnetostrictive coefficient with decreasing temperature. While in the first stage, the MC, MS and the ME^{therm} anisotropy are in the same order than the ME^{RT}

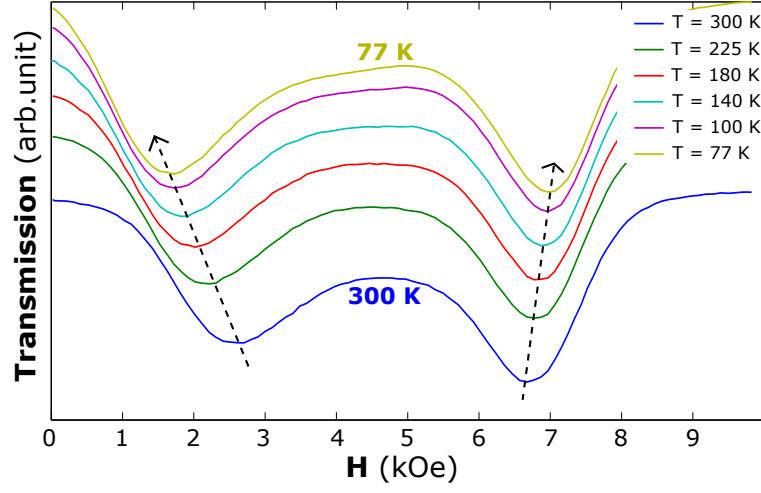


Figure 6.12 – Absorption spectra for the first 21 nm-diameter Ni nanowire array at 30 GHz at different temperatures, from room temperature to 77 K. Dashed arrows are guide for the eyes to show the shift of both absorption peaks with the temperature. Vertical translation of curves is applied for clarity.

	Low H_r	High H_r
K_{eff} [$10^5 \text{erg} \cdot \text{cm}^{-3}$]	19.7	7.61
K_{ms} [$10^5 \text{erg} \cdot \text{cm}^{-3}$]	5.4	
K_{mc} [$10^5 \text{erg} \cdot \text{cm}^{-3}$]	-2.05	
K_{me}^{therm} [$10^5 \text{erg} \cdot \text{cm}^{-3}$]	-1	
K_{excess} [$10^5 \text{erg} \cdot \text{cm}^{-3}$]	17.4	5.3

Table 6.6 – Resonance conditions and characterization of energy from anisotropy contributions at 77 K for 21 nm-diameter Ni nanowires in AAO membranes.

	Second stage	First stage
ϵ	-0.95 %	-0.25 %

Table 6.7 – Strains for the two-stages system in 21 nm-diameter Ni nanowires at 77 K.

contribution, so the positive anisotropy induced by the lattice mismatch is counterbalanced by the negative MC and ME^{therm} contributions. All these energies will induce a small positive shift in the resonance field at 77K, as shown in the calculation (table 6.6).

6.4.3 Additional experiments

In this section, we will present the results of additional experiments performed either on other small diameter Ni nanowire arrays (i. e. pH solution, $\varphi = 35$ nm, nanowires length), either by changing some fabrication parameters (i.e. electrodeposition potential) or other nanoporous templates (i.e. polycarbonate).

35 nm-diameter Ni nanowire arrays

Shifts in resonance fields are also been observed in other Ni nanowire samples embedded in alumina membrane with higher pores diameter. Fig. 6.13 shows the absorption curves of 35 nm diameter Ni nanowire samples electrodeposited with different pH electrolytic solutions. It has already be shown that the acidity of the electrolytic solution influences the crystallinity of the Ni material [123, 124] due to the larger hydrogen formation in the low pH electrolytic solution which prevents a good crystallinity. In Fig. 6.13, we compare the FMR absorption spectra for 35 nm-diameter Ni nanowire samples fabricated with different pH solutions, from 1.5 to 5. Typical Ni nanowire samples are made using an electrolytic solution with pH = 3.7. All the samples have nanowires length higher than 60 % of the membrane height.

In Fig. 6.13, the dark long-dashed line is the sample made with the normal pH solution (pH = 3.7) used for Ni nanowire samples previously presented in this chapter. A shouldering effect, located at the right of the main peak, is observed on this curve. The proximity between resonance field of the main peak and the shoulder indicates that the values of strain in both stages of the nanowire are slightly similar. Thanks to the formula in table 6.1, these strain values can be calculated and are equivalent to $\epsilon_2 = -0.6$ % for the second stage and to $\epsilon_1 = -0.26$ % for the first stage. Compressive strain in the second stage is then smaller for 35 nm-

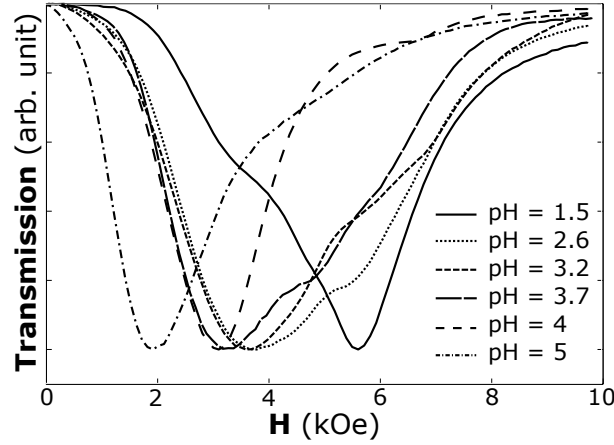


Figure 6.13 – Transmission field sweep at 25 GHz for Ni-AAO 35 nm-diameter nanowire samples made with different pH electrolytic solutions.

diameter nanowires than the 18 nm- and 21 nm-diameter Ni nanowire arrays, which can be seen in the XRD measurements as the decrease of the crystallinity of larger diameter Ni nanowires, while in the first stage, the compressive strain has a similar value in the 35 nm-diameter sample than in the 18 nm and 21 nm-diameter nanowire samples, which can be interpreted as a similar crystallinity in the first stage in small diameter Ni nanowires.

In Fig. 6.13, the dependence of the FMR absorption on the pH of the electrolytic solution is obvious. Shift of the resonance field to lower magnetic field is observed when the pH value increase from 1.5 to 5. For the nanowire sample made with pH = 1.5 (solid line), the value of the strain corresponding to the main FMR peak and calculated using the energy considerations becomes negligible, while for the sample with pH = 5, the calculated strain corresponding to the main FMR peak is $\epsilon = -0.95\%$. The increase of the pH value of the electrolytic solution originates an highly enhancement of the crystallinity of the Ni nanowire, but also the decrease of the length of the first stage.

Fig. 6.14 shows the absorption spectra for various Ni nanowire samples made with different velocities of the nanowires growth and having different lengths of nanowires. The pH value of the electrolyte is 3.7. The electrodeposition voltage $E = -1.1$ V (solid and long-dashed lines - typical used potential) leads to an axial growth rate of 14 nm/sec. Lower rate could be reached by applying lower intensity of voltage. Indeed, $E = -0.9$ V (short-dashed and dotted lines) induces an axial growth rate

of 2.5 nm/sec and $E = -0.8$ V (dashed-dotted line) a rate of 0.5 nm/sec. Fig. 6.14 compares also the results obtained for samples with different nanowires lengths.

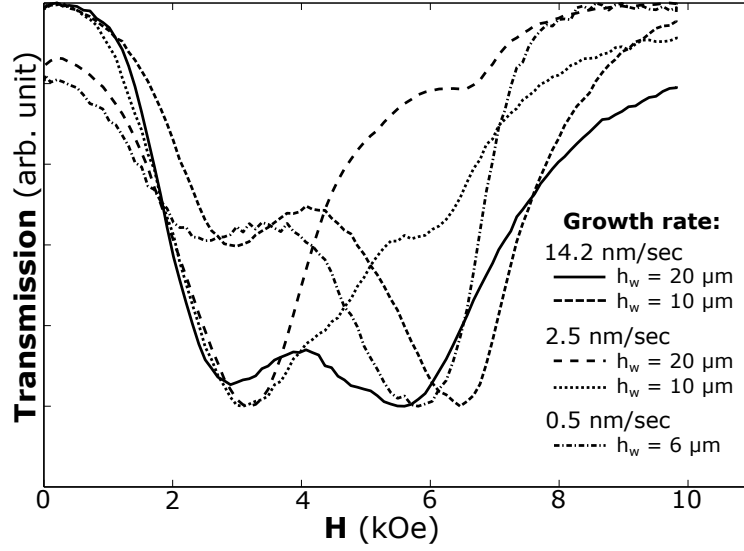


Figure 6.14 – Transmission field sweeps at 25 GHz for Ni-AAO 35 nm-diameter nanowire samples with different axial growth rates and nanowires lengths.

A double FMR peak (or shoulder) structure appears in each absorption spectrum with the low resonance peak below 4 kOe, and the high resonance peak above, which can be considered as the second stage or the first stage contributions, respectively. Taking into account only the axial growth rate for similar nanowires length samples, lower velocities of nanowires growth seems to influence the length of the first stage: for samples with $h_w = 20 \mu\text{m}$ (solid and long-dashed lines) or for samples with $h_w = 10 \mu\text{m}$ (short-dashed and dotted lines), the lowest growth rate (dashed-dotted line) shows a higher intensity for the high resonance field, which means that the length of the first stage is larger than the second one. In the case of the nanowires lengths characterization, longer nanowires have an increased ratio of the absorption intensity between low resonance and high resonance peaks, corresponding to an increase of the volume ratio of the second stage when the nanowires length increases. A characterization of larger numbers of samples could bring higher statistics over dependence of the strain on the growth rate or on the nanowires length.

Small diameter Ni nanowires in PC templates

In Fig. 6.15, the absorption spectra for Ni nanowire arrays in polycarbonate membranes shows small shift in the resonance field and small shouldering effect in the small diameter (40 nm) arrays compared with the large diameter (200 nm) nanowire array. The difference between the porosities (2.6 % for small diameter and 5.5 % for large diameter) is not enough to explain the shift in the resonance magnetic field. Vertical lines in Fig. 6.15 indicate the theoretical resonance magnetic fields calculated by $H_r = f_r/\gamma - 2\pi M_s(1 - 3P)$.

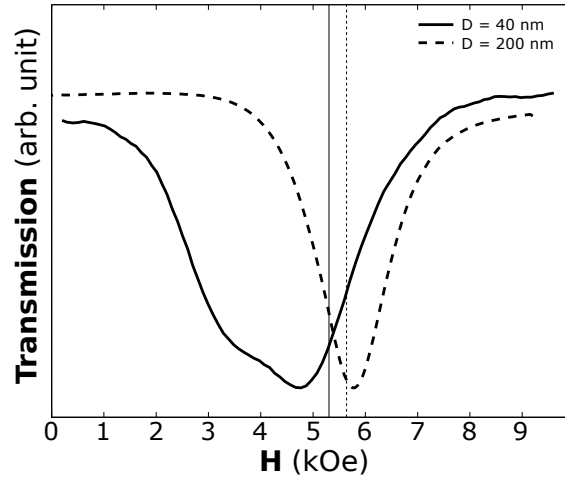


Figure 6.15 – Absorption spectra at room temperature for Ni nanowire arrays in nanoporous polycarbonate membranes at 25 GHz with pores diameter and porosity : $\varphi = 40$ nm and $P = 2.5$ %, and $\varphi = 200$ nm and $P = 5.5$ %. Vertical lines indicate the theoretical resonance magnetic field calculated considering only the MS contribution.

Considering the measurements at room temperature of small diameter Ni nanowires in a polymer membrane, energetic characterizations using the formula in table 6.1 indicate the strain of the observed resonance absorption. 40 nm-diameter Ni nanowires in PC membrane can be compared to the 35 nm-diameter in AAO membrane where a compressed strain value $\epsilon = -0.6$ % was calculated. A smaller strain ($\epsilon = -0.15$ %) is calculated for Ni -PC sample. It could result due to the fact that the polymer membrane is much softer than the alumina and then the compressive stress is slightly compensated by the compression of the polymer membrane due to the induced stress applied by the wire.

6.4.4 Another hypothesis : core-shell system

In addition to the two-stages system, another geometry system could be considered: the core-shell system (Fig. 6.16). This hypothesis implies that the core of the nanowire has a low (or zero) deformation of the Ni lattice and the shell has a relatively high deformation due to the lattice coefficients mismatch between alumina and Ni. A dependence of the width of the shell δ on the z -axis parallel to the wire axis provides the dependence of the contribution from each part on the length of the nanowires: the width of the shell δ increases with the length of the nanowire.

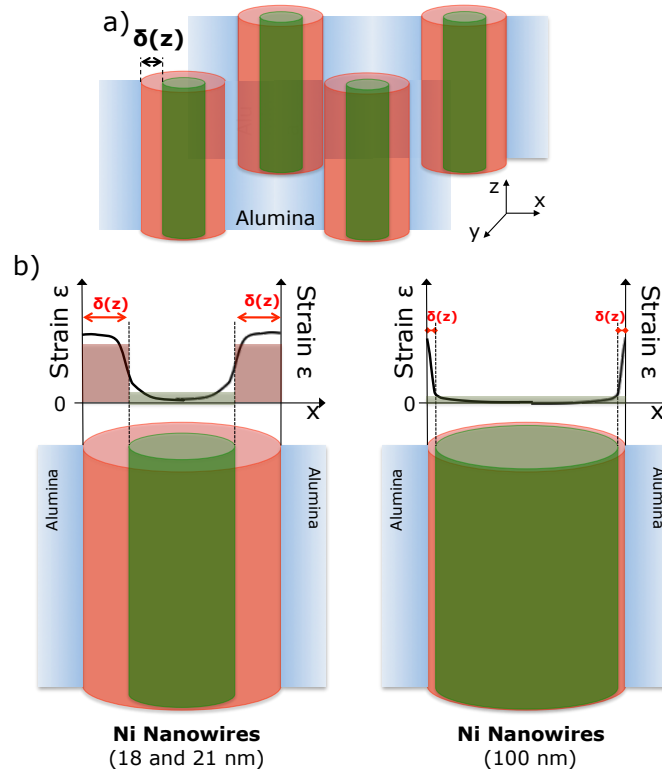


Figure 6.16 – (a) Sketch of core-shell system representing the small diameter Ni nanowires embedded in alumina membrane. The core (green part) is not deformed, while the shell (red part) is deformed. Shell has a width δ depending on the z -axis position. (b) Sketch of the two-systems core-shell geometry with the schematics of the induced strain ϵ of both systems. An average strain can be calculated for both parts.

Fig. 6.16(b) shows sketches of the core-shell system for Ni nanowires

with different diameters. In the case of small diameter Ni nanowire (18 and 21 nm), as the FMR measurements indicate, the volume fraction of each system is quite equivalent (21 nm) or larger for the shell (18 nm). The width of the shell δ evolves slightly with the z-axis. If we consider that the average width is $\delta = 6$ nm, the volume ratio is similar for the both systems in the case of 21 nm-diameter samples, and 1/3 - 2/3 for core - shell system respectively, in the case of 18 nm-diameter sample.

The calculated data indicate that a compressive stress takes place on the small diameter nanowires shell, corresponding to a strain of about 1 % for the 18 nm and 21 nm sample. The non-zero values of the strain on the high resonance absorption peaks corresponding to the core contribution for 18 and 21 nm-diameters samples could be explained by a residual strain inside the core of the wire induced by the large strain existing in the shell (Fig. 6.16 (b)). An average value of strain can be found for both core-shell systems thanks to the energetic consideration: $\epsilon_{shell} = 1$ % and $\epsilon_{core} = 0.2-0.3$ %. Due to the small proportion of the shell ($\delta \ll \varphi$) in the 100 nm-diameter Ni nanowire array, the average strain on the nanowires can be neglected.

The main issue of this hypothesis is originated from the rapidly drop of the strain on the border of the core-shell system. As demonstrated in [125] for Ni film grown on Cu single crystal, the strain could be maintained for a certain width before rapidly dropping. An average strain could be found for each system.

6.4.5 Conclusions

Series of FMR measurements done on small diameter Ni nanowire arrays in AAO templates have shown two-phases behavior distributed in a two-stages geometry. Large shifts of the FMR absorption and the apparition of a double peak (or shoulder) configuration on the field sweep at room temperature indicate the presence of two different preferential anisotropies in Ni nanowires. In the first stage, the crystallographic structure of Ni nanowires is weakly ordered, causing a small deformation in the lattice, while in the second stage the Ni has a highly crystallographic structure oriented in the (110) direction in the wire axis. The lattice of this second stage is compressed due to the lattice coefficient mismatch between Ni and alumina or due to a friction along the pores wall during the electrodeposition process. This increase of the crystallinity in small diameter samples has been shown in the XRD measurement. The two-stages geometry induces a wider hysteresis loop in the case of small diameter nanowire samples due to the enhanced magnetic anisotropy in the second stage, and a single reversal magnetic field appears for each

monodomain nanowire.

Energy considerations for the different magnetic anisotropies have given the value of the structure deformation for the two stages. These values of deformation in the case of Ni-AAO with small diameter are equivalent to the values obtained for Ni-PVDF template at low temperature. Even this energetic calculation could explain the different behavior in temperature of both FMR peak observed in the 21 nm-diameter Ni nanowire array in an AAO template. These opposite shifts of FMR absorptions have been explained by the temperature dependence of the magnetostriction coefficient for Ni and by the fact that the energy contributions are quite different for both FMR absorptions.

Besides, some additional experiments have been done to characterize the fabrication and understand the mechanism that gives deformation to the second stage of nanowires. The experimental comparison of samples made with different pH value electrolytes has demonstrated the influence of the crystallinity of the Ni on the deformation of the wire. Less acid will be the electrolytic solution, higher will be the crystallinity and larger will be the shift of the resonance field. A characterization of the axial growth rate and the length of nanowires confirms the two-stages configuration in small diameter Ni nanowire. The length of the first stage is dependent on the growth rate. Indeed, slower rate would reduce the length of the first stage where the Ni lattice is not highly oriented. Longer lengths of nanowires would contribute to reinforce the contribution of compressed second stage in the FMR absorption. Small shouldering effects have been observed in small diameter Ni nanowires in PC membranes. These results could be attributed to two-stages geometry, with the specification that the softness of the PC membrane reduces the deformation of the Ni structure in the second stage.

Further measurements have to be done to complete the explanation of this effect, by identifying the right hypothesis and the exact size of the two-systems geometry. A deep characterization of the nanowires length could be done to understand the dependence of the FMR contribution of the deformed-nondeformed systems. Finer XRD measurements could be done to observe the deformation in the Ni structures.

We should also think about the mechanisms that induces the compression of the Ni structure in the second mono-crystalline stage of the wire. A model for the electrodeposited nanowires should be proposed as a function of the pH, the voltage and the rate of the growth.

Chapter 7

A template approach for novel magnetic-ferroelectric nano-composites

7.1 Introduction

The abilities to tune the properties of nanowire arrays through their interactions with the host matrix is of importance. A change in the magnetic properties of Ni nanowire arrays embedded into polycarbonate or PVDF membranes has been reported at low temperature (see section 6.3) as a result of the large thermal expansion coefficients mismatch between the polymer membrane and the Ni nanowires [66, 67, 68]. However, microwave devices operating in a temperature-controlled environment are far from adequate since they may require robust and energy-intensive cryogenic systems. In the other hand, changes in magnetic properties of Ni nanowires are also been observed at room temperature for small diameter Ni nanowire arrays embedded in nanoporous alumina membranes (see section 6.4) or with smaller intensity in polycarbonate membranes [126]. Nevertheless, the misunderstanding of the occurring magnetic anisotropy gives some difficulties to develop a tunable microwave device. Therefore, it is of paramount importance to enable external control of the matrix-dominated nanocomposite magnetic properties in order to design frequency agile microwave devices operating at room temperature.

In this chapter, we present a new route to control the magnetic properties of such arrays of ferromagnetic nanowires by electrochemical pore-filling of ferroelectric polyvinylidene difluoride (PVDF) polymer tem-

plates rich in polar β form, thus making multiferroic nanostructured composites. In such two-phase composite materials, a magnetoelectric effect may arise from a mechanical coupling between the magnetostrictive and piezoelectric phases. Indeed, the application of electric field induces a stress on the piezoelectric constituent that is passed onto the magnetostrictive ferromagnet. It should be mentioned that evidence for multiferroic behavior was previously demonstrated in multilayered composite material made of PVDF and ferromagnetic layers [71]. In order to enhance this coupling the interfacial area between the two phases should be maximized. This can be accomplished by considering rationally designed nanoscale composites based on nanowires, which have a large surface to volume ratio. We report on experimental studies by ferromagnetic resonance (FMR) at room temperature for the magnetoelectric effect of Ni nanowires embedded in PVDF membranes. COMSOL Multiphysics was used to simulate the magnitude and spatial distribution of the electric field surrounding individual Ni nanowires in the array. In both transmission line configuration, shear piezoelectric effects are found from simulations in the PVDF matrix surrounding the lateral surface of the nanowires and are most probably associated with the weakening of the magnetic anisotropy along the nanowires easy axis, as observed experimentally.

7.2 Materials and microwave design

7.2.1 Samples used in the present study

We used commercially available 28 μm -thick PVDF films to produce track-etched ferroelectric polymer membranes using a similar process as the PC membrane [87]. The PVDF film was uni-axially stretched and poled by the producer prior to the track-etching process. The pore size and pore density are 105 nm and $2 \cdot 10^8 \text{ cm}^{-2}$, respectively. For comparison, 22 μm -thick PC templates of similar porosity were also made. A method of measurement has been applied to obtain spatial distribution of the electric field inside the membrane by applying an adjustable bias voltage of 200V through the electrode pads on the transmission line structure. Both positive and negative voltages were applied on the transmission line while the ground electrode was maintained at zero potential.

7.2.2 Stripline design

In the stripline geometry measurement, a 500 μm -width stripline line is evaporated on the other side of the cathode (Fig. 7.1). A positive or

negative voltage is applied on this stripline while the cathode is maintained at zero potential. A pronounced tip effect is observed in this kind of transmission line device. Another coplanar lines configuration has been suggested to reduce the issue. These results are presented in appendix A.

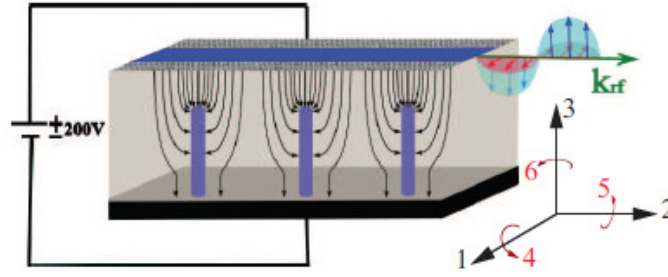


Figure 7.1 – Schematic description of the stripline transmission line consisting of a metallic ground plane (and cathode), a ferroelectric PVDF polymer template filled with magnetic nanowires and the 500 μm wide metallic strip line. An adjustable bias voltage up to ± 200 V is applied through the two electrodes pads. The calculated electric field lines due to this bias are indicated, as well as definition of piezoelectric axes.

7.3 Numerical simulations of the spatial distribution of electric field

A finite element method with COMSOL Multiphysics has been used to simulate the magnitude and spatial distribution of the static electric field from the bias voltage surrounding individual Ni nanowires in the array. In Fig. 7.1, the calculated electric field lines are also shown. The electric field surrounding the end of each nanowires is strongly enhanced by the tip effect and radially distributed field lines appear in the neighborhood of the nanowire apex. Below the nanowire tip, the electric field is mostly aligned perpendicular with respect to the nanowire axis. In Fig. 7.2 (a)-(b), the calculated spatial variation of the electric field intensity is shown considering separately the vertical component (i.e. the axial direction of the nanowire) and one component of the field in the plane of the PVDF film for a normalized filling height $h_{\text{Ni}} = 0.5$ of the electrodeposited Ni nanowires. The packing factor P of the array was fixed to 0.16% and the applied voltage was 200 V. The calculations reported in Fig. 7.2(a) show that the field penetration between parallel standing

nanowires is poor due to the proximity between neighboring nanowires so the large height of the nanowires compared to interwire distance lead to an electrostatic screening effect. As shown in Fig. 7.2(b), the electric field is mostly directed perpendicular to the nanowire axis in the region surrounding the lateral surface of the nanowires. It is most intense at the tip and its magnitude decays rapidly underneath the nanowire extremity. As expected, the field intensity increases in the upper part of the nanowires as the relative height of the nanowires is larger, e.g. $h_{\text{Ni}} = 0.9$, as shown in Fig. 7.2(c).

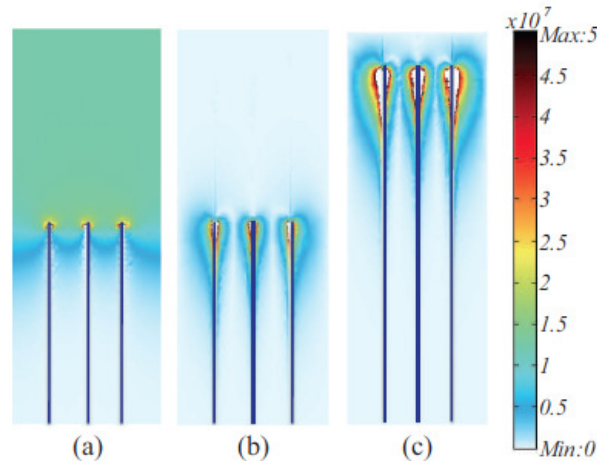


Figure 7.2 – Spatial distribution of electric field intensity in a PVDF - Ni nanowired nanocomposite ($P = 0.16\%$, $h_{\text{Ni}} = 0.5$) considering separately (a) the vertical component and (b) one component of the field in the plane of the PVDF film; (c) same as in (b) with $h_{\text{Ni}} = 0.9$. The color scale represents the magnitude of the field in V/m.

It is interesting to note that in the region surrounding the lateral surface of the nanowires, the electric field has a large component perpendicular to the PVDF polar axis, denoted as "3", which is perpendicular to the surface of the film, see Fig. 7.1. Therefore, shear occurs and the large shear piezoelectricity components d_{15} and d_{24} in PVDF relate the strain induced by the perpendicular electric field as follows, $\epsilon_5 = d_{15}E_1$ and $\epsilon_4 = d_{24}E_2$. In the above notations, the stretching direction of the film is denoted as "1". The "2" axis is orthogonal to the stretching direction in the plane of the film. The shear planes, indicated by the subscripts "4", "5", are perpendicular to the directions "1" and "2" [127]. The inset of Fig. 7.3 shows a schematic representation of the shear strain in the PVDF matrix close to the nanowires tip, with displacements of

the polymer surface along the polarization direction and no piezoelectric deformation along the electric field distribution. From the quantitative determination of the electric field distribution surrounding the nanowires we may calculate the resulting shear strain ϵ_5 . We used $d_{15} \sim -25$ pC/N as a typical value for uni-axially stretched PVDF film [128]. In Fig. 7.3, the magnitude of the shear strain ϵ_5 in the PVDF matrix surrounding the lateral surface of the nanowires is reported along the nanowire axis for 3 different nanowires packing P values, respectively 0.016% (continuous line), 0.16% (dotted line) and 1.6% (dash-dotted line). In all cases, the shear strain is at its maximum at the extremity of the nanowires. Furthermore, the decay length of the shear strain along the axis of the nanowire depends strongly on the nanowire packing. It varies from ~ 2 μm for $P = 1.6\%$ up to almost the entire length of the nanowires for $P = 0.016\%$.

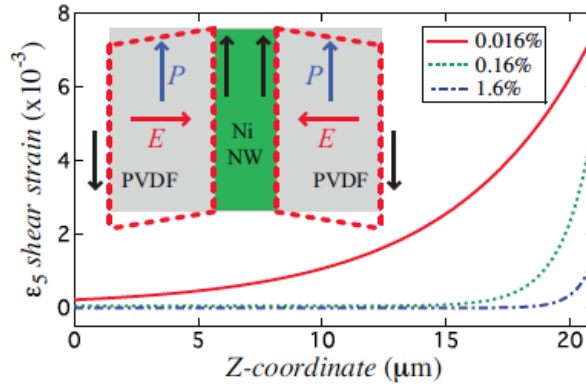


Figure 7.3 – Variation of the calculated shear strain ϵ_5 in PVDF matrix surrounding the lateral surface of the nanowires for 3 nanowire packing densities, $P=0.016\%$, $P=0.16\%$ and $P=1.6\%$, respectively. In all cases, the relative height of the Ni nanowires, $h_{\text{Ni}} = 0.75$. The inset shows a schematic representation of the shear strain appearing in the PVDF matrix at the interface with a Ni nanowire with the black arrows indicating the strain orientation.

7.4 Evidence for magnetoelectric effect in PVDF-Ni nanocomposites from FMR measurements

Fig. 7.4 shows representative FMR transmission spectra recorded at 25 GHz in an array of Ni nanowires ($\varphi = 105$ nm), by applying the external magnetic field parallel to the nanowires. Results are shown for

applied voltages of ± 200 V as well as without bias electric field. At zero bias voltage, a good agreement is found for the resonance field H_r considering the simple formula $H_r = f_r/\gamma - H_{ms}$ where $H_{ms} = 2\pi M_s(1 - 3P) \sim 2.9$ kOe is the magnetostatic field including both shape and dipolar interaction terms [23], f_r is the resonance frequency, $\gamma = 3.09$ GHz·kOe $^{-1}$ and $M_s = 485$ emu·cm $^{-3}$ are the gyromagnetic ratio and the saturation magnetization for Ni, respectively. Very similar shifts in resonance field towards higher magnetic field of about 160 Oe are observed both for positive and negative voltages. Such an increase in resonance field ΔH_r corresponds to a decrease in the resonance frequency $\Delta f_r = \gamma \Delta H_r \sim 500$ MHz, and is consistent with a reduction in uni-axial magnetic anisotropy. This decrease is observed experimentally in the effective permeability of the nanowired substrate extracted from transmission and reflection measurements [129] in frequency-sweeping mode, for various values of *dc* biasing magnetic field: a shift of its FMR frequency towards lower frequencies occurs when a bias voltage is applied. FMR voltage tuning of permeability is present even in absence of *dc* magnetic field. This is of prime interest for reconfigurable devices involved in wireless transceivers and antennas arrays: voltage-controlled oscillators (VCOs), circulators, differential phase-shifters, etc., could be tuned between 5 GHz and 50 GHz depending on the nature of the ferromagnetic nanowires fixing FMR and on values of *dc* biasing voltage. As expected, no shift in resonance field has been observed in a PVDF-Py sample (see inset in Fig. 7.4), which is ascribed to the fact that permalloy has negligible magnetostriction [67, 130]. It should be also emphasized that no *E*-field induced effects were found in PC-Ni samples in which the polymer matrix shows no piezoelectricity.

Even if the exact nature of the stress that is imposed on the nanowires remains to be determined, it can be replaced by an equivalent field, H_{me} , the magnetoelastic component of the effective field, and will be of the order of [116, 73]

$$H_{me} = \frac{3\lambda E \epsilon}{2M_s}, \quad (7.1)$$

where λ is the magnetostriction coefficient ($\lambda \sim -30 \cdot 10^{-6}$ for polycrystalline Ni nanowires), E is the Young modulus on Ni (200 GPa) and ϵ denotes the strain. The experimental values for the shift in resonance field measured in several PVDF-Ni samples are in the range 40 - 160 Oe. Using Eq. (7.1), the changes in effective field correspond to strains in the range $2 - 8 \cdot 10^{-4}$, which is consistent with the calculated values reported in Fig. 7.3 for the case $P = 1.6\%$. From the simulations of Fig. 7.3, it appears that for more diluted nanowire arrays, e.g. $P =$

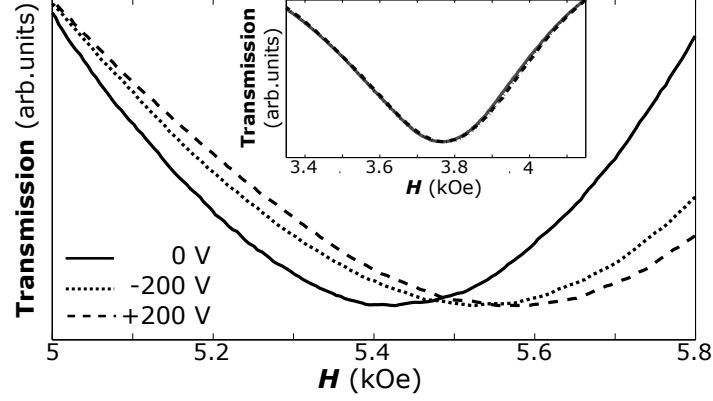


Figure 7.4 – Field sweep microwave absorption spectra recorded at 25 GHz with the field applied parallel to the nanowires for an array of Ni nanowires ($\varphi \sim 105$ nm, $h_{\text{Ni}} \sim 0.75$ and $P = 1.6\%$) embedded in a PVDF membrane. Experimental data shown for zero bias voltage applied (continuous line), as well as for non-zero values ($V = +200$ V, dashed line; $V = -200$ V, dotted line). The inset shows similar FMR transmission spectra for a PVDF-Py sample with $\varphi \sim 105$ nm and $P = 1.6\%$.

0.016%, not only the strain is much larger at the tip (by a factor 5), but also it expands over whole length of the nanowire, so that one can anticipate that strongly enhanced shifts in resonance frequency, up to several GHz for the same applied voltage, should be observed. However, such small nanowires concentrations fall below the sensitivity threshold of conventional transmission line based FMR measurements.

Furthermore, it should be emphasized that the effects slightly depend on the polarity of the bias voltage, although the magnitude of the shift in resonance field towards higher magnetic field for a selected sample may differ up to 25% at $V = +200$ V and $V = -200$ V. A detailed analysis of the FMR results would require a refined theoretical model for the mechanical stress introduced by the piezoelectricity of PVDF and the resulting strain in the Ni nanowires. This is however outside the scope of the present chapter.

7.5 Conclusions

We investigated a new route to control at room temperature the magnetic properties of nanowire arrays by fabricating Ni nanowires into track-etched ferroelectric PVDF nanoporous templates. A stripline con-

figuration for applying the voltage inside the nanowired substrate is used to study numerically the distribution of electric field and experimentally observe the dependence of the magnetic properties on the applied voltage.

A high electric field up to $5 \cdot 10^7 \text{ V} \cdot \text{m}^{-1}$ is observed in the surrounding of the nanowires apexes but the penetration of electric field inside parallel standing nanowires is poor due to an electrostatic screening effect. All along the nanowire, electric field is perpendicular to the wire. The screening effect leads to a decrease of the shear strain along the nanowires and this decay is much more pronounced for high porosities, for example, for $P = 1.6 \%$, the $21 \mu\text{m}$ -long nanowires experience non-negligible shear strains only on the $2 \mu\text{m}$ -length close to its apex.

FMR measurements are done without voltage and with applied $\pm 200 \text{ V}$ voltage using the stripline configuration. We observe a small shift to higher resonant magnetic field, which can be linked to a decrease of the magnetic anisotropy due to an axial tensile stress applied to the Ni nanowires. The strain calculated thanks to the experimental measurements is in the same order that the strain calculated from numerical predictions.

The fabrication of magnetostrictive non-conductive nanowire arrays, such as ferrite material, should greatly improve the control of the magnetic properties by applying a *dc* electric field. In the stripline configuration, it would be able to avoid completely the screening effect and diffuse a parallel electric field all along the nanowires, which experience a right strain due to the stress induced in the template by the electric field.

General Conclusion

The main part of this work has been devoted to the conception and the fabrication of microwave devices based on ferromagnetic nanowire arrays embedded in self-supported nanoporous alumina membranes. Three devices based on different topologies have been fabricated and experimental results proved the promising alternative to the ferrite materials for communication systems. High observed working frequency ranges and their self-biased behavior allow to dedicate these devices to integrated airborne or spatial applications. Modeling and parametric characterizations predicted resulting performances that meet the commercial standard requirements and confirm the high potential of the nanowired substrates for on-board systems. In the second part of the thesis, we provided a deep study on the characterization of magnetoelastic anisotropy in these nanocomposite systems and their possible development in a microwave device, using a magnetic-ferroelectric nanocomposite. We report parametric dependences of the magnetoelasticity either at low temperature or room temperature and more detailed model were proposed for observed enhanced anisotropy in small diameter Ni nanowire arrays.

First, modelings of intrinsic properties of a nanowired substrate have been proved to agree with experimental results. The determination of these properties is crucial to tune the properties of microwave devices based on nanowired substrates. The decrease of the insertion losses of microwave devices is the main issue to meet the required performances. A characterization of the dielectric losses of AAO membranes suggests that a drying time of 4h to reduce the residual moisture inside the pores is not enough to reduce the insertion losses, and that amorphous AAO membranes have lower dielectric losses than α - or γ -alumina. The thickness of transmission lines has to reach at least $2\text{ }\mu\text{m}$ to avoid the eddy current or a high roughness of the line. NiFe and CoFe nanowire arrays have a good remanence and stability in temperature, which allows to develop integrated devices for aerospace and airborne applications.

Concerning the nonreciprocal microwave devices based on the microstrip topology, we first demonstrated experimentally the large differential phase shift ($> 300^\circ/\text{cm}$) observed in high frequency range (30 - 50 GHz) and without any external applied magnetic field. The edge-guided mode principle and the specific topology of nanowired substrate including two magnetic alloys inside parallel areas induce the nonreciprocal effect, as explained in the theoretical model which ensures good agreements between numerical predictions and experimental results. Due to the relatively-high dielectric losses of the commercial AAO membrane, insertion losses inside the devices are large (20 dB) with the consequence that the figure of merit has a low value ($15^\circ/\text{dB}$). Theoretical predictions of optimized nanowired substrate and topology show that this figure of merit can increase up to $500^\circ/\text{dB}$. Moreover the differential phase shift value is tunable with several geometric aspects of nanowired substrates, as the width of each nanowires zones or the nanowires lengths. This tunability gives the possibility to find a microwave gyration ($\pm 180^\circ$) for each frequency bands required in aerospace domains, with the required performances in term of insertion losses. Microwave isolator can also be developed using high nanowires length for both magnetic materials. The signal will flow in the forward direction of propagation while it will be stopped in the other. In this case, a high isolation (60 dB) is visible only for the frequency corresponding to the FMR frequency of CoFe nanowires.

The three-ports junction topology is the second nonreciprocal microwave device developed in this thesis. Both three-ports and two-ports configurations can be applied to this device. The principle based on the counter-rotating mode in cylindrical cavity is widely dependent to the intrinsic effective dielectric and magnetic properties of the substrate. A highly-fine dependence of the circulation frequency is demonstrated and modeled on the nanowires lengths. Planar microwave circulators working at dedicated frequencies in the X -band were fabricated in the purpose to integrate airborne applications. NiFe nanowire arrays have demonstrated circulations in the X -band with value larger than one required for commercial standard (15dB), while CoFe nanowires arrays have shown low remanence state which avoid the nonreciprocal effect. This issue has been solved using commercial AAO membranes with smaller pores diameter and porosity. High dielectric losses present in commercial AAO membranes increase the insertion losses of the devices, which prevent the performances to achieve the required standards. Low-loss dielectric membrane and adapted access lines dimensions numerically succeeded

to reduce these insertion losses lower than 2 dB. Two-ports isolators have been fabricated on the three-ports junction topology, by applying a short-circuit to the third port. Numerical parametric characterization allows finding microwave isolations for desired frequency bands used in aerospace applications and isolations have been experimentally observed in three frequency bands, using either NiFe or CoFe nanowires.

The ferromagnetic nanowire inductor is a compact planar reciprocal device that can be integrated in a circuit. We proved the compatibility of the nanowire array embedded in an AAO membrane with microlithography techniques used in integration of devices for systems in package or on chip. The high aspect ratio of nanowires, coupled with the high packing fraction of the array, enables to supply a large amount of magnetic material to maximize the value of inductance while the nanowired AAO membrane limits substrate losses up to several GHz and avoid eddy current. We experimentally demonstrated the improvement of the quality factor up to 7 GHz in the case of nanowired substrates, due to the increase of the inductance value and the limitation of the increase of losses. The equivalent circuit of planar inductors on nanowired substrates provides the theoretical basis for the model, which agrees with the experimental results. Thanks to the increase of the magnetic volume inside the substrate, the improvement of the quality factor can be maximized up to 35% by an optimization of the pores filling, and the working frequencies can be increased up to 14 GHz by applying a external magnetic field perpendicular to the substrate. The enhancement of capacitance value for the nanowired substrate compared to the empty AAO membrane proves the validity of the model of the effective permittivity for large packing fraction nanowired substrate. This improvement of the quality factor has been shown for a wide range of meander or spiral inductors with various dimensions.

The characterization of magnetic anisotropies for specific ferromagnetic nanowire arrays provided main information about properties of these nanowired substrates and their possible conceptions of microwave devices. Magnetoelastic effects seen at low temperature on Ni nanowire arrays embedded in polymer templates as PC and PVDF induce a large increase of the magnetic anisotropy of these nanowired substrates. In such composite systems, the mechanical coupling arises at low temperature from the thermal coefficient mismatch between Ni nanowire and polymer membrane and becomes larger than the shape anisotropy. This increase induces a large shift in the FMR field and therefore causes a change in the microwave behavior of the nanowired template. Shifts of

the resonance field larger than 6 kOe have been measured in samples constituted by Ni nanowires inside nanoporous PVDF membrane of porosity 1.7 %. The corresponding shift of the resonance frequency is about 16 GHz. A reverse behavior has been observed when heating the nanowired samples, which confirms that a tensile stress induced on the nanowires by the membrane decreases the anisotropy. A model permits to calculate the value of the magnetoelastic energy induced in these systems and to compare with other energy contributions. In the case of Ni nanowires in low porosity PVDF membrane, we saw that the magnetoelastic energy contribution is much larger at low temperature than other contributions, while in the case of large diameter Ni-AAO samples, magnetoelastic energy has the same value of other contributions. An estimation of the magnetoelastic anisotropy can be made using the calculated strain induced on the nanowires, taking into account the membrane material, through its hardness, and its porosity. The developed model showed good agreement with experimental data of various membrane materials and porosities nanowired samples.

As a second characterization of the magnetoelastic anisotropy, we compare the experimental results obtained for Ni nanowires arrays with different diameters, where an enhancement of the anisotropy is observed for small diameter samples. We proposed as detailed hypothesis a two-stages system existing in small diameter Ni nanowires. Ni nanowire includes two phases distributed along the wire. The first phase can be seen as the nucleation of the Ni nanowire at the bottom of the pore where the crystallinity of the Ni is low and no deformation is observed in the Ni lattice, and the second one represents the second part of the wire where the crystallinity of Ni in the (110) direction is high and a deformation of the wire is observed. These two phases can be seen in the double absorption peak configuration measured in small diameter Ni nanowires. A magnetoelastic anisotropy can exist at room temperature in Ni nanowires due to the specific process of electrodeposition and to the hardness of alumina. XRD measurements confirm the high crystallinity of the small diameter Ni nanowires that axially grow with the (110) crystallographic direction. The no-observation of these two-phases structures in the hysteresis loop could be explained by the dynamic magnetization reversal of a wire that prevents a double states magnetization. Energy considerations indicate a compressed strain in the second phase structure $\epsilon = 1\%$ in the case of very small diameter Ni nanowires (18, 21 nm), which is a typical value of strain measured at low temperature in Ni-PVDF samples. We also observed a dependence on the nanowires length of the phase contributions, which implies a constant length for the first non-deformed phase. Further studies have to be done to con-

firm this dependence. Using electrolyte with lower pH prevents a high crystallinity of the Ni and reduces the enhanced magnetoelastic anisotropy as we observed in the experimental results. The two-phases system is inhibited. Two-phases systems is also present in small diameter Ni nanowires embedded in PC membrane, with the difference that the softness of PC membrane compensates the stress in the nanowires.

Magnetoelectric effects present a new route to control the magnetic properties of ferromagnetic nanowire arrays. Using ferromagnetic resonance measurements under static voltage bias, we demonstrated a magnetoelectric effect arising from a mechanical coupling between the magnetostrictive and piezoelectric phases. The calculated electric field lines and intensity indicate that PVDF matrix surrounding the surface of the Ni nanowires experiences shear stress. The competing magnetoelastic anisotropy originating from the piezoelectric effect leads to a reduced anisotropy field along the wire axis. The nanowires packing in the array are found to play a dominant role in the magnetoelectric effect.

In conclusion, the development of microwave devices using magnetic nanowired substrates would allow to solve the main issues of classical microwave ferrites, i.e. the enlargement of the working frequencies up to 50 GHz, coupled with the miniaturization of the devices by an integration in a circuit. Large reciprocal and nonreciprocal effects observed in our devices compete with the one proposed in the literature. The knowledge of the magnetic anisotropy contributions in such nanocomposites, as the magnetoelastic effect at low and room temperatures, enlarge the possibility to conceive devices with tunable working frequencies.

As perspectives to this work, one should continue the researches to improve the transmitted signal of microwave devices to reach the standard requirements of industry. It might be accomplished looking for reducing the insertion losses either by a fabrication process of lower dielectric loss AAO membranes, or by an optimization of all the nanowired devices, including transmission lines. One should also develop a process of fabrication that permits to integrate the nanowired substrates in a circuit.

Many other microwave applications may be develop using nanowired templates, as a *nanowire waveguide* or a *leaky-wave antenna*.

Nanowire waveguides are made by a specific configuration of the nanowired area inside the AAO template. Full-filled pores metallic nanowires are used as the vertical walls of a wave-guide. Transmission lines deposited on both sides of the nanowired substrates serve as the horizontal walls. The high dielectric constant of the AAO membrane improves the

transmitted signal and the small thickness of the nanowired substrate allows to integrate the device in a circuit. Next, the electrodeposition of magnetic nanowires in an area parallel and at a certain distance to the walls achieves the nonreciprocal behavior of the nanowire waveguide. Preliminary fabrication test and measurements are already done to prove the feasibility of the waveguide with a nanowired substrate.

The leaky-wave antenna uses quite similar waveguide structure for emitting signal with high directivity. The numerical behavior of this device is already proved in [34] and parametric characterization is done.

In the case of magnetoelectric effects of the Ni nanowires, further experiments have to be done to understand the specific two-stages process of electrodeposition for small diameter Ni nanowires embedded into AAO or PC membranes. This understanding would benefit to all electroplated magnetic nanowires. In the nanowires-PVDF substrate, the fabrication of magnetostrictive isolating nanowire arrays, such as ferrite material, should greatly improve the control of the magnetic properties by applying a *dc* electric field.

Appendix A

Coplanar configuration for novel magnetic-ferroelectric nanocomposites

This section reports the magnetoelectric effect of coplanar configuration for transmission line design. To avoid the large tip effect that appears in the stripline configuration, we conceived a coplanar lines design, which should homogeneously distribute the electric field in the nanowired substrate. An electric field would be applied between the transmission line and grounds of the coplanar structure etched in the gold layer of the sample. Some nanowires grown in the PVDF template touch the transmission line or the ground and could serve also as electrodes to apply a diffusive electric field in almost all the membrane height (Fig. A.1). This configuration allows to reduce the tip effect by applying an electric field in the plane of the substrate. We should deduce that all nanowires lying in the gap between coplanar lines are under an electric field in the perpendicular direction of the nanowires axis.

A.1 Coplanar design fabrication

The conception of the coplanar lines used a new technique of fabrication for microwave devices based on a nanoporous membrane : instead of depositing a transmission line on the other side of the cathode, we etch this cathode layer using the laser micromachining system to remove the gold lying in the gap between microstrip and grounds. This technique has already been used to fabricate coplanar lines or circulator based on a nanowire array embedded in a nanoporous alumina membrane that could resist to high temperature. In the case of a nanoporous polymer

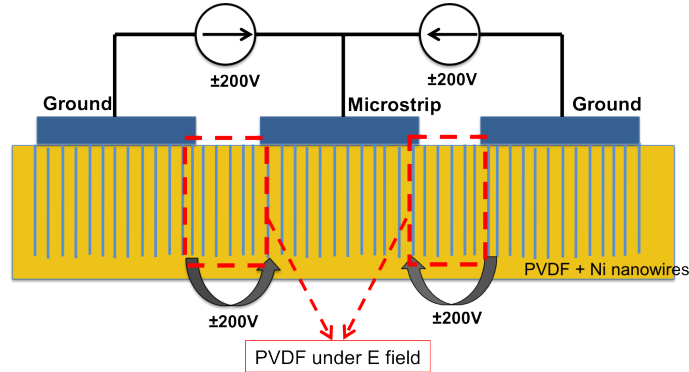


Figure A.1 – Schematic description of the coplanar lines consisting of a metallic transmission lines and two grounds etched in the cathode layer of the sample. An adjustable bias voltage up to ± 200 V is applied between the transmission line and grounds. Nanowires lying below the transmission lines or below the grounds could also be used as electrodes to apply a homogeneous electric field in all the membrane height.

template, the difficulty stays in the fact that we should find the best parameters of etching to remove all the gold without burning the polymer substrate. Fig. A.2 shows two pictures of the extremities of the same coplanar lines based on a Ni nanowire array embedded in a PVDF membrane. Geometric parameters of the nanowire array are a length of 70% of the height of the membrane and a packing factor of 5%. As these images prove, the etching of the gold layer with the laser micromachining system is quite uncertain as the left-side extremity of the coplanar line shows a good etching, while the gold layer is not totally etched in the right-side extremity. This could be the consequence of a high roughness due to the use of a GaIn eutectic paint for the electrodeposition in this kind of membrane. As the process of etching is done in one step, etching the remaining gold layer is not possible since the laser will burn the already revealed PVDF membrane. Despite the hazardous behavior of the etching, we could measure this sample by cutting the unetched part of the coplanar lines. The sample length is then 0.7 cm.

A.2 Numerical spatial distribution of *dc* electric field

A finite element method with Comsol Multiphysics has also been used to simulate the magnitude and spatial distribution of the static

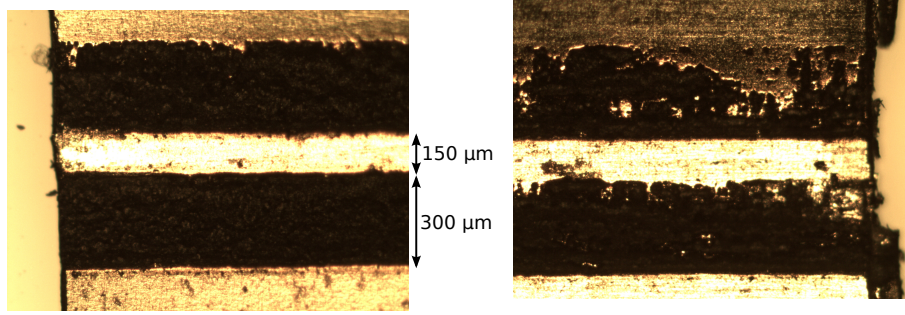


Figure A.2 – Microscope images of the etched coplanar lines of the PVDF polymer template including Ni nanowire array of packing factor $P = 5\%$ and nanowires length $h_{Ni} = 70\%$. Both images represent each other the extremities of a coplanar line with a width of $150\ \mu\text{m}$ for the transmission line and $300\ \mu\text{m}$ for the gap between transmission line and grounds, and the length of coplanar lines are 1 cm. Yellow parts are the gold layers.

electric field from the bias voltage surrounding individual Ni nanowires. A numeric sample is made by aligning Ni nanowires between the ground and the transmission line with a linear packing density of $P = 0.16\%$. The symmetry of the grounds allows calculating only one part of the coplanar lines. The gap width is $w = 300\ \mu\text{m}$, the ground and transmission line thickness is $h_l = 5\ \mu\text{m}$, the membrane height is $h_p = 30\ \mu\text{m}$ and the nanowires length is $h_{Ni} = 15\ \mu\text{m}$. Fig. A.3 shows a slice perpendicular to the nanowires axis and situated at the middle of the nanowires length from a top view of the nanowired substrate and represents the calculated dc electric field components perpendicular to the nanowires axis and to the transmission line (x -component) and along the nanowires axis (z -component) for an applied bias voltage of 200 V. As expected, the electric field in the surrounding of the nanowires shows high planar-direction components. The calculation reports a difference in the electric field surrounding nanowires close to the transmission line or to the ground and in the middle of the gap. The divergence reaches two order of magnitude and an opposite mirror symmetry exists taking an imaginary line passing by the middle of the gap. This difference is the consequence of a tip effect created by the thin layer of the transmission line or the ground. Interestingly, the x -electric field surrounding each nanowire has an opposite direction at the right-side of the nanowire than at the left-side. As expected and similarly to the stripline configuration, the vertical component of the electric field is zero although it exists a small tip effect at the apexes of the nanowire. Another impor-

tant difference in the electric field surrounding nanowires close to the transmission line or close to the ground is the opposite direction of the field: close to the transmission line, the electric field has a direction toward the nanowires, while near the ground, the direction of electric field goes out the nanowires.

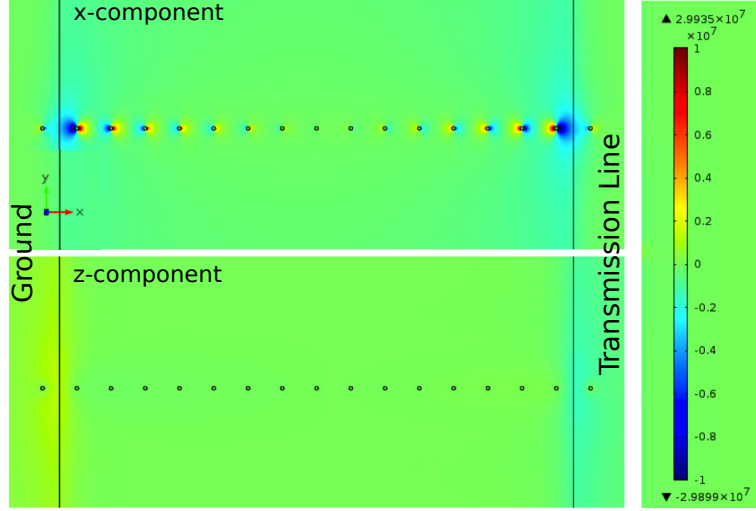


Figure A.3 – Top view of the spatial distribution of the electric field inside the PVDF-Ni nanowires nanocomposite ($P=0.16\%$ and $h_{Ni} = 50\%$) considering separately (a) the x -component perpendicular to the nanowires axis and to the transmission line and (b) the vertical z -component parallel to the nanowires axis for a planar slide located at the middle of the nanowires length. The color bar represents the magnitude of the field in V/m.

The spatial distribution of electric field surrounding the nanowires is similar to the stripline configuration. We could calculate the shear strains $\epsilon_5 = d_{15}E_x$ and $\epsilon_4 = d_{24}E_y$ using the same formula and taking the typical value for uni-axially stretched PDVF film $d_{15} \sim -25$ pC/N [128]. Fig. A.4 (a) represents the shear strain in the PVDF matrix surrounding the nanowires all along the nanowires length for the cases of a nanowires lying close to the transmission line (blue line), in the middle of the gap (green line) or close to the ground (red line). In a first observation, the tip effect present in the stripline configuration disappeared and all the length of the nanowires is strained with the same strength. This force is smaller of one order of magnitude than the maximum strain existing in the stripline configuration for a porosity of 0.16 % (see Fig. 7.3). As a second observation, the shear strain applied on the nanowires in the middle of the gap is negligible, as expected from

the magnitude of electric field. As a last information, shear strains that nanowires close to the ground feel are the opposite than the one close to the transmission line feels. In Fig. A.4 (b), we observe the shear strain all along the circumference of the nanowires at mid-length, as explained in the Fig. A.4 (c). We take as hypothesis that $d_{24} = d_{15}$. x -direction and y -direction represents ϵ_5 and ϵ_4 respectively. The most important information of this graph is that the global shear strain (sum of both components) is not the same in the entire circumference.

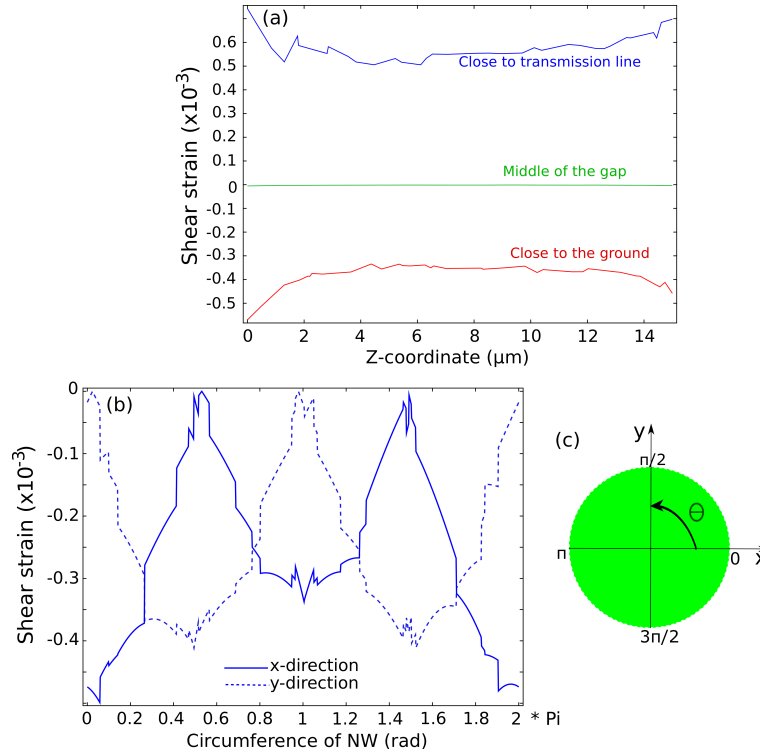


Figure A.4 – Shear strain in the PVDF matrix (a) in the surrounding of all the nanowires length for nanowires lying close to the transmission line (blue curve), in the middle of the gap (green curve) and close to the ground (red curve); and (b) in the surrounding of the circumference of a nanowire, as explained in (c).

Because of the wide gap ($w = 300 \mu\text{m}$) between transmission line and ground, the value of the electric field inside a PVDF matrix without any nanowires is weak ($E = V/w \sim 6 \cdot 10^5 \text{ V/m}$). To increase this value, we did some additional simulations with several gap widths ($w = 300 \mu\text{m}$, $100 \mu\text{m}$ and $20 \mu\text{m}$) and several porosities ($P = 0.16 \%$ for $300 \mu\text{m}$ and $100 \mu\text{m}$, and $P = 1.6 \%$ for $20 \mu\text{m}$). Fig. A.5 shows the shear strain in the

PVDF matrix surrounding the nanowires all along the nanowires length for different widths of the gap. We can conclude that, at this scale, the shear strain is not dependent on the gap width or on the porosity. Even the change of conductivity for nanowires does not affect the intensity of electric field in the surrounding of the nanowires since Ni, Al or Cu nanowires have experienced similar shear strains.

Coplanar configuration allows avoiding the high electric tip effect present in the stripline configuration but with the main drawback that the behavior of the electric field is different beside all nanowires lying in the gap between the transmission line and the grounds. Even around one single nanowire, the electric field does not have the same intensity. Before the misunderstanding of the effect of positive or negative strains on the magnetic properties of Ni nanowires, we should take carefully all the information from any experimental measurements.

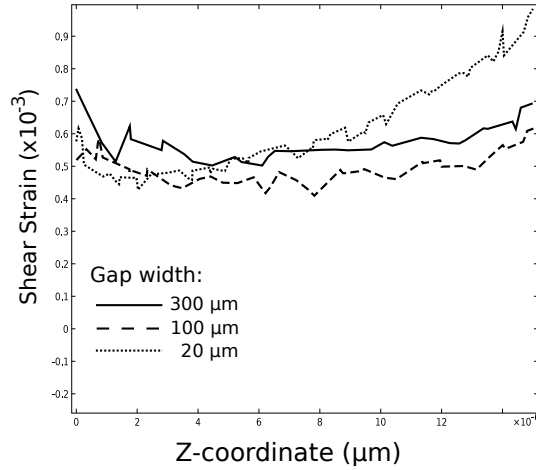


Figure A.5 – Shear strain in the PVDF matrix all along the nanowires length for different widths of gap and porosities: $w = 300 \mu\text{m}$ and $P = 0.16 \%$, $w = 100 \mu\text{m}$ and $P = 0.16 \%$ and $w = 20 \mu\text{m}$ and $P = 1.6 \%$.

A.3 Experimental confirmation

Experimental measurements have been realized on the 0.7 cm length coplanar lines (Fig. A.2) based on a Ni nanowires array of packing density $P = 5\%$, nanowires diameter of $\varphi = 150 \text{ nm}$ and relative nanowires lengths $h_{\text{Ni}} = 0.7$. We first check the stability of the sample by measuring the dispersion relation from the magnetic field sweep at different

frequencies to prove that the Ni nanowire array has not been damaged by the etching process. The relation dispersion is in good agreement with the formula $H_r = f_r/\gamma - H_{ms}$ where $H_{ms} = 2\pi M_s(1 - 3P) \sim 2.6$ kOe. A first observation informs that the FMR absorption is very weak due to a small amount of ferromagnetic nanowires involved in the microwave excitation. Fig. A.6 shows a representative FMR transmission spectra

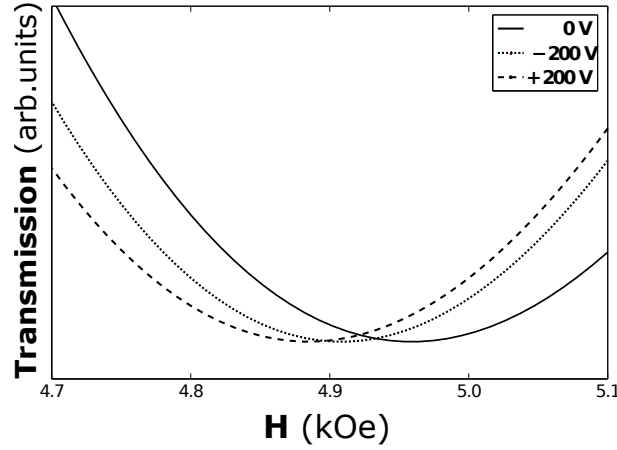


Figure A.6 – Field sweep microwave absorption spectra recorded at 25 GHz with the field applied parallel to the nanowires for an array of Ni nanowires ($\varphi = 165$ nm, $h_{Ni} \sim 0.7$ and $P = 5.5$ %) embedded in a PVDF membrane with a coplanar lines configuration. Experimental data shown for zero bias voltage applied (continuous line) field, as well as for non-zero values ($V = +200$ V, dashed line; $V = -200$ V, dotted line).

recorded at 25 GHz in this sample, by applying the external magnetic field parallel to the nanowires. Results are shown for applied voltages of ± 200 V as well as without bias electric field. Small similar shifts are observed in the case of negative voltage ($\Delta H \sim -50$ Oe) or in the case of positive voltage ($\Delta H \sim -80$ Oe). The weak FMR absorption brings high uncertainty in the measurements and similar field sweep at different constant frequencies does not show a shift in the FMR frequencies between measurements with applied voltage and without voltage. Nevertheless, small negative shifts in the range -50 to -130 Oe are observed for a large number of frequencies. It is worth mentioning that the porosity of this coplanar lines sample ($P = 5$ %) is higher than the stripline configuration ($P = 1.6$ %), but the independence on the porosity of the calculated shear strain along the nanowires should keep constant the observed shift in the case of lower porosities. The shift to lower resonant magnetic fields should be the consequence of an enhancement of the magnetic anisotropy

of Ni nanowires.

A.4 Conclusions

The coplanar configuration of the PVDF-Ni nanowire composite does not reach the expected performances. Since this structure might avoid the tip effect present in the nanowires apexes in the stripline configuration, the present sample has numerically shown misunderstanding distribution of electric field surrounding the nanowires. No similar shear strain are induced in all the nanowires lying into the gap of coplanar lines, even more, the electric field intensity is different around one single nanowire. Experimental measurements have shown small negative shifts in the FMR absorption for the sample under bias positive or negative voltages, which should assume an enhanced anisotropy and then a small axial tensile strain of the Ni nanowire.

The uncertainties in the distribution of electric field, as well the difficulties of processing the coplanar lines on a polymer membrane, are great issues, which requires stopping the characterization of this type of configuration. As announced in the perspectives, the fabrication of magnetostrictive isolating nanowire arrays, such as ferrite material, should greatly improve the control of the magnetic properties.

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