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Self-biased coplanar circulator based on electrochemically grown ferrimagnetic nanowires

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Abstract:

This paper is about a self-biased coplanar circulator. The design approach is based on the use of magnetic composite substrates self-polarized made from ferrimagnetic nanowires. It consists of integrating and orienting cobalt ferrite (CoFe₂O₄) nanocrystallites in porous alumina membrane using the magneto-electrodeposition technique. XRD, SEM and EDX analysis confirmed the formation of the spinel phase of cobalt ferrite with a good distribution of Co, Fe and O elements in the unit cell of CoFe₂O₄ nanowires. The performed SQUID-VSM measurements have revealed that the nano-composite electrodeposited and cooled under a magnetic field of 0.6 T has the best magnetic properties, i.e. a large anisotropy field (H_a), moderate coercive field (H_c), and partial squareness ratio (M_r/M_s). This nano-composite has been selected as an interesting candidate for the fabrication of a miniaturized self-biased circulator. The study has started with the implementation of an analytical dimensioning process based on Bosma's theory. Based on interesting HFSS simulation results, a first prototype was fabricated in the clean room and characterized at high frequencies. The measurements of the S parameters have highlighted a circulation phenomenon of 3 dB.

Keywords: porous alumina membrane; CoFe₂O₄ nanowires; self-biased circulator; HFSS; circulation phenomenon; S parameters

1. Introduction

The microwave devices are currently used for applications in many industries, from mobile telephony to aerospace sectors and radar systems. In recent years, their development has rapidly progressed due to increasing demand, has led engineers to enlarge the frequency bands usually and has looked for advanced miniaturization of the devices, through circuit integration among other the ability to master the magnetic

properties of the materials making up these devices. In this respect, the electromagnetic metamaterials, defined as effective structures with properties generally not available using conventional materials, and designed from elements whose size is much smaller than the guided wavelength [1], form a promising class of materials for the telecommunications sector. Several works have been published on innovative research on metamaterials [2-6], but very few magnetic materials had been involved in the design of metamaterials at microwave frequencies. However, metamaterials can produce non-reciprocal effect without adding active devices [7]. In other hand the magnetic materials exhibit a high magnetic activity at microwave frequencies [8]. Depending on the nature of the material and the experimental conditions, a magnetic material subjected to a static and dynamic magnetic field can exhibit a gyromagnetic resonance response at high frequencies. Several microwave devices based on magnetic materials, such as circulators, make use of the gyromagnetic resonance of magnetic materials, by operating out of to the resonance frequency band. This type of device is often used in the systems with antennas [9,10], The circulator is a device non-reciprocal, typically having three ports. Basically, a device is non-reciprocal if the transmission of the wave differs according to the directions of propagation of the device. The conventional circulator operates with ferrite substrates polarized by an external magnetic field to ensure the non-reciprocity of the electromagnetic signals propagation. However, the presence of bulky and heavy permanent magnets that ensures the polarization of the ferrite remains a major obstacle to miniaturize and to integrate this circulator in microelectronic circuit. However, their integration into planar devices requires overcoming some technological challenges [11]. The nanowires technology offers an interesting alternative for massive and bulky ferrites for the fabrication of self-polarized planar devices. Several prototypes of microwave devices based on ferromagnetic nanowires have been demonstrated. Microstrip circulators have been constructed by Saib et al, [12] Allaeyes et al, [13] and more recently by Darques et al.[14] Circulators asymmetrically charged with nanowires have been designed in microstrip configuration by Spiegel et al.[15] and in coplanar guide configuration by Kuanr et al. [16] Carignan et al. reported a circulator in isolator mode at double-band displacement, in microstrip line configuration, using the double resonance of ferromagnetic nanowires [17]. De La Torre Medina et al. manufactured a circulator in phase shifter mode based on nanowires [18]. Stripline circulators have been reported by Spiegel et al. [19] and Hsu et al. [20] In 2011, Boyajian et al. [21] produced a prototype of a coplanar circulator. The magnetic substrate that has been used consists of magnetic particles of barium hexaferrite mixed in SU-8 resin matrix deposited by technical spin coating on an alumina substrate. During the magnetic substrate growth process, a permanent magnet was placed under the alumina substrate in order to orient the magnetic moments of the particles. The measurements showed low reciprocity caused by wrong orientation particles and lower concentration. In the same context, Ardaches et al. [22] tried to develop a self-biased coplanar circulator. The approach chosen is based on the synthesis of nano-ferrimagnetic composite

substrates. It consists of making a deposit by dip-coating of CoFe_2O_4 nanoparticles into porous alumina membranes. He manufactured and characterized his coplanar circulator at high frequencies. The measurement has shown a circulation phenomenon, which remains very low due to the small concentration of the magnetic nanoparticles in the composite.

This type of circulator is still under study because it should allow to avoid using permanent magnets and to reduce the costs with simple and collective manufacturing techniques. Since the performance of circulators based on magnetic materials is based on the ability to control the magnetic response of the magnetic material, the new approach is to fabricate a self-polarized ferrimagnetic nanowire substrate. Among the techniques for synthesizing magnetic nanowires, we can cite RF sputtering, plasma vapor deposition, laser pulse deposition, etc [23]. These techniques, which are still under development, are very expensive, require sophisticated equipment [24]. Many research centers are interested in processes to obtain low-cost self-polarized materials. The electrochemical deposition technique is considered to be a very promising, simple and cheaper alternative for the manufacture of self-polarized nanowires. This paper is devoted to the magneto-electrochemical growth of CoFe_2O_4 ferrimagnetic nanowires, due to its high magneto-crystalline anisotropy and magnetization saturation and its physico-chemical and microwave characterization. We focus on the modelling and fabrication of a coplanar circulator based on the synthesized auto-polarized ferrimagnetic nanowires. Firstly, the permeability tensor model of the nanowires will be presented. Afterwards a first circulator sizing based on the Bosma's theory is obtained, followed by a numerical modelling of the coplanar circulator on the HFSS software. Then, several structures will be simulated in order to determine which one gives the best performances. Finally, the manufacturing steps of such component on the nanowires surface will be discussed. **Afterwards, a microwave characterization will be performed for the fabricated circulator.**

2. Experimental

2.1. Synthesis procedure

Analytical grade iron sulfate and cobalt sulfate purchased from Sigma Aldrich company were used as starting precursors. The electrochemical growth tests were carried out using a Teflon conventional cell of three electrodes connected to a Potentiostat VoltaLab 40 PGZ 301 controlled by a computer via VoltaMaster 4 software. Anodized aluminum oxide (AAO) membranes of 20 nm in pore diameter, 50 μm in thickness, $10^9 \text{ pore}/\text{m}^2$ in porosity, 1 cm^2 of area and metallized by 3 μm of copper layer were used as working electrodes (Fig. 1).

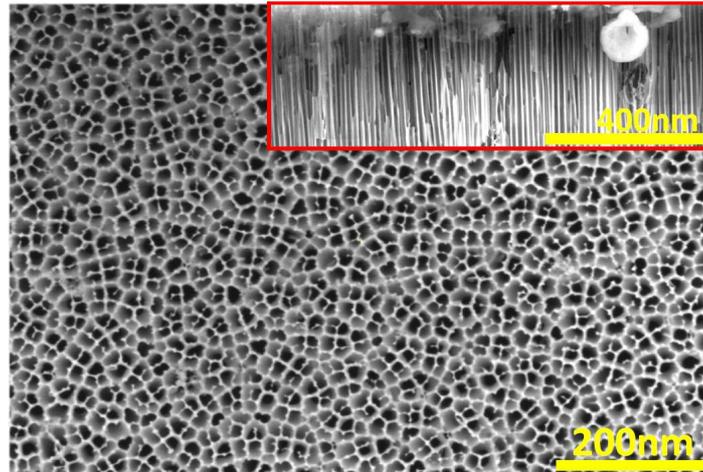


Fig. 1. Top and cross sectional view SEM images of AAO membrane.

A Platinum rod and a silver/silver-chloride (Ag/Ag-Cl) were used as the counter electrode and as the reference electrode, respectively. During the electrodeposition experiments, a perpendicular applied magnetic field, generated by a commercial NdFeB magnet (30 mm in diameter and 15 mm in height) is placed under the AAO membrane substrate in order to enhance the electrochemical reaction of the ions reduction. The CoFe_2O_4 nanowires were electrodeposited from an electrolyte that containing 0.06 M of $\text{Fe}(\text{SO}_4)\cdot 7\text{H}_2\text{O}$, 0.04 M of $\text{Co}(\text{SO}_4)\cdot 7\text{H}_2\text{O}$. In order to adjust the pH value at 3, 0.04 M of sulfuric acid (H_2SO_4) was added to the electrolyte. Before starting the electrochemical deposition, the AAO membranes substrates were ultrasonically cleaned with acetone for 10 min each. Next, the Co and Fe ions were reduced by applying a voltage of -1.2 V vs the working electrode. Thus, the as-prepared nanowires were annealed in air at 500 °C for 24 h and then magneto-cooled at 0.6 T in order to complete the crystallographic formation process and obtain a pure spinel phase of CoFe_2O_4 .

2.2.Characterization

The chronoamperometry technique was exploited to study the electrochemical deposition evolution of the Co and Fe elements. The microstructure of the synthesized nanowires was investigated by recording the X-ray diffraction (XRD) spectrum using a PAnalytical X'Pert PRO X-ray diffractometer that operating with Cu-beam ($\lambda = 0.154$ nm). Then, the morphology and the elemental composition of CoFe_2O_4 nanowires were studied using Quanta 200-FEI scanning electron microscope (SEM) that operated at 40 kV and equipped with energy dispersive X-ray detector (EDX). Moreover, the surface topography and roughness have been estimated by a Bruker Dektak XT profilometer. In addition, the hysteresis loop was evaluated by a superconducting quantum interference device (SQUID) magnetometer. Finally, the numerical and experimental S-parameters, of the etched coplanar circulator on the nanowires surface, were investigated using high frequency structure simulator (HFSS) and RS ZVA67 vector network analyzer (VNA).

3. Results and Discussion

3.1. Microstructural, morphological, and magnetic properties

The chronoamperometric study offers an idea of the mechanism of nucleation and growth of CoFe_2O_4 inside the AAO membrane pores. From this study, it is possible to determine the required time to completely fill the pores of the nanoporous membranes and also to determine the critical overflow time of the deposit.

Fig. 2 shows the chronoamperogram recorded during the electrochemical growth of CoFe_2O_4 within 20 nm-AAO membranes at an applied potential of -1.2 V. It can be observed from the recorded curve that in the early stages of growth when the diffusion layer is formed, the pores start to be filled by CoFe_2O_4 nanocrystallites. This is translated by practically quasi-static current density. In addition, the deposition is maintained for a duration of up to 40 minutes, in which the porous nanowires become completely filled.

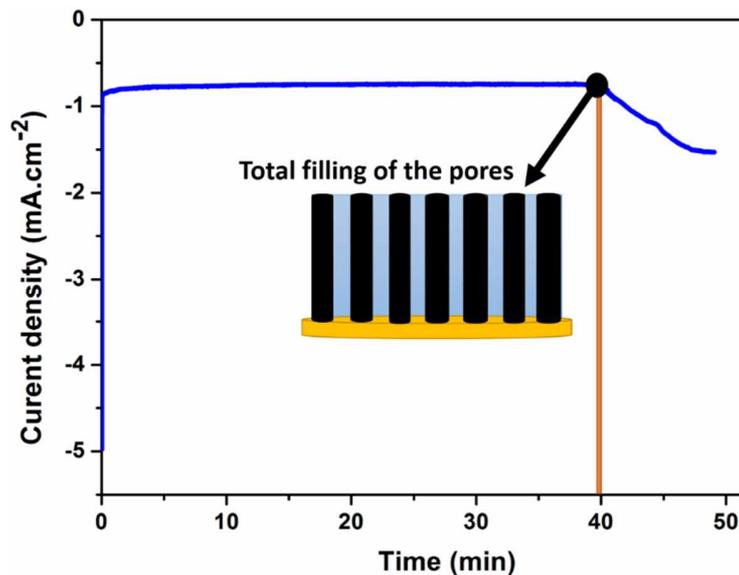


Fig. 2. The chronoamperogram of the magneto-electrochemical growth of CoFe_2O_4 into nanoporous AAO membrane at room temperature.

Finally, the current density continues to **negatively** increase and the deposit continues to accumulate on the surface of the nanoporous membrane. From this result, it is assumed that the deposition time is fixed at 38 min in order to prevent the presence of a top layer that can influence the measurement and fabrication processes that will be presented in the next sections.

To examine the crystal structure of the obtained nanowires after being annealed at 500 °C for 24 hours and magneto-cooled at 0.6 T, X-ray diffraction spectrum was recorded. Fig. 3 shows the X-ray diffraction reflections of the grown nanowires in an AAO membrane at room temperature. As observed, the XRD peaks correspond to the CoFe_2O_4 spinel structure (JCPDS n. 22-1086) [25]. We note the absence of any kind of

secondary impurities. The Al_2O_3 peaks originate from the alumina substrate. The lattice parameter a of CoFe_2O_4 which is crystallized in face-centered cubes (fcc), was determined by an average on all intense peaks. The calculation provided a value of 8.38 \AA . This value is in accordance with bulk material CoFe_2O_4 [25].

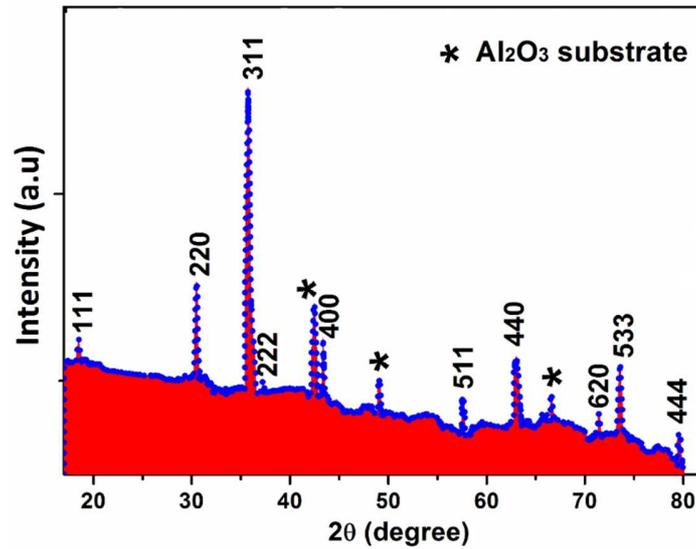


Fig. 3. XRD spectra of elaborated CoFe_2O_4 nanowires.

Moreover, the morphology of the synthesized nanowires has been studied using SEM microscopy. Before starting the surface scanning, the nanowired substrate is immersed in a diluted iron perchloride and sodium hydroxide (NaOH) solutions in order to remove the copper serving as cathode and the alumina. As shown in Fig. 4, the top view image revealed that the electrodeposited CoFe_2O_4 nanowires are completely filled and have an average diameter that is varying between 20 and 24 nm. These values are approximately equal to the pores diameters of the AAO template. Concerning the chemical composition, the EDS measurements indicate the existence of Co, Fe and O. In addition, no copper peak is detected, confirming that the copper film is completely removed by the iron perchloride. The absence of any impurities in the EDS examination confirms the high purity of the synthesized nanowires. From the collected percentages values, it can be concluded that the average atomic ratios of O, Fe and Co are 57.85 %, 25.62 % and 16.53 %, respectively. As a result, the obtained nanowires present a high stoichiometry of O, Co and Fe elements, which indicate the good distribution of their cations in the spinel structure of CoFe_2O_4 .

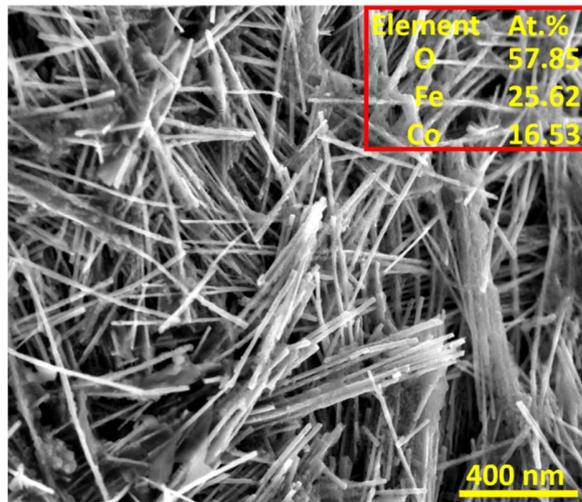


Fig. 4. SEM image of CoFe_2O_4 nanowires and its elemental composition.

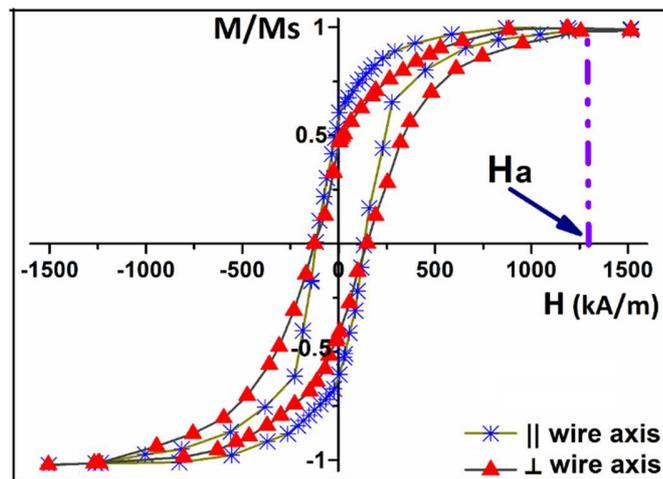


Fig. 5. Room-temperature hysteresis loop of CoFe_2O_4 nano-composite.

The magnetic properties of CoFe_2O_4 nanowires are examined by a SQUID magnetometer. During the magnetic scanning, the magnetic field was applied parallel to the wire axis. The deposition time was interrupted at 38 min to avoid the formation of any kind of thin layer on the nano-composite surface. The measured hysteresis loops at 300 K for CoFe_2O_4 is shown in Fig. 5. The measurements clearly indicate that the elaborated nanowires array present a ferrimagnetic behavior [25]. From the recorded loop, it is noted that the nanowires possess a squareness ratio (M_r/M_s) of 0.6, a coercive field (H_c) of 140 kA/m and an anisotropy field of 1.5 T. This result confirms the elaboration of partially hard ferrimagnetic substrate with high shape anisotropy and magnetic internal magnetic field of CoFe_2O_4 crystallites. According to the magnetic response of CoFe_2O_4 nanowires, a coplanar circulator based on these auto-polarized nanowires has been designed.

3.2. Application of CoFe₂O₄ nanowires

After examining the magnetic properties of CoFe₂O₄ nano-composite synthesized by the magneto-electrodeposition technique, an analytical modeling and electromagnetic simulation of the Y-junction circulator has to be made. A theoretical model used to predict the permeability tensor of CoFe₂O₄ nano-composite in non-saturated state (remanence state) has been defined. Tests will be performed using different squareness ratios (m) for CoFe₂O₄ nano-composite. Afterwards, we will discuss more concretely the phenomena involved in the circulator. We note that the circulator has been primarily studied in its stripline configuration. In our work, a first approach uses the theoretical study of the stripline circulator based on Bosma's formulas to find the approximate circulator dimensions. Then, the calculation of the S-parameters, taking into account the different loss factors, will make it possible to evaluate the performance of the circulator and to transpose its dimensions towards the coplanar topology. On the other hand, a protocol of integration for the theoretical model of the magnetic permeability in the Ansoft HFSS commercial software will be followed in order to numerically simulate the structure previously studied in an analytical approach. Indeed, the 3D electromagnetic simulation enables the ferrite thickness to be taken into account, which is not the case in the first analytical modeling.

Finally, best resulting design will be used for the manufacturing of a typical prototype.

3.2.1. Magnetic permeability tensor of CoFe₂O₄ nano-composite

Electromagnetic models of unsaturated ferromagnetic nanowires leading to the permeability tensor are often complex. A permeability model on Ni, CoFeB and Ni/Cu nanowires has been developed by Carignan et al. [17, 26], but it is applied on metallic ferromagnetic nanowires and it should be modified for application on ferrite nanowires. The tensor permeability model must take into account the response of the nanowired ferrimagnetic composite with their magnetic characteristics such as M_s , M_r/M_s , H_i and geometrical ones such as the porosity p and the filling rate h_w of the AAO membrane pores. In this first approach the interactions between the nanowires and the interactions between the domains into each nanowire are neglected. Owing to these simplifications, the modified Irashagi model permeability tensor model of the nano-composite has been selected [27,28].

$$\hat{\mu}_{wires} = \begin{bmatrix} \mu & -j\kappa & 0 \\ +j\kappa & \mu & 0 \\ 0 & 0 & \mu_z \end{bmatrix} \quad (1)$$

Where

$$\mu = 1 + 2\pi\mu_0 M_s \gamma p h_w (f_r + jf\alpha) ((f_r + jf\alpha)^2 - f^2)^{-1} (m^2 + 1) \quad (2)$$

$$\kappa = 4\pi\mu_0 M_s \gamma p h_w f ((f_r + jf\alpha)^2 - f^2)^{-1} m \quad (3)$$

$$\mu_z = 1 + 2\pi\mu_0 M_s \gamma p h_w (f_r + jf\alpha) ((f_r + jf\alpha)^2 - f^2)^{-1} (1 - m^2) \quad (4)$$

By way of illustration, consider the following conditions: $\mu_0 M_s = 0.18 T$, $f_r = 42 \text{ GHz}$, $\alpha = 0.017$, $p = 50 \%$ and $h_w = 100 \%$.

Some results of this model with an orientation of magnetic crystallite moments varying from 30 % to 100 % have been studied. Figs 6, 7 and 8 show the variations of the components of μ , κ and μ_z corresponding to the permeability tensor as a function of the average of the moments orientation ($m = M_r/M_s$) of the CoFe_2O_4 nanocrystallites.

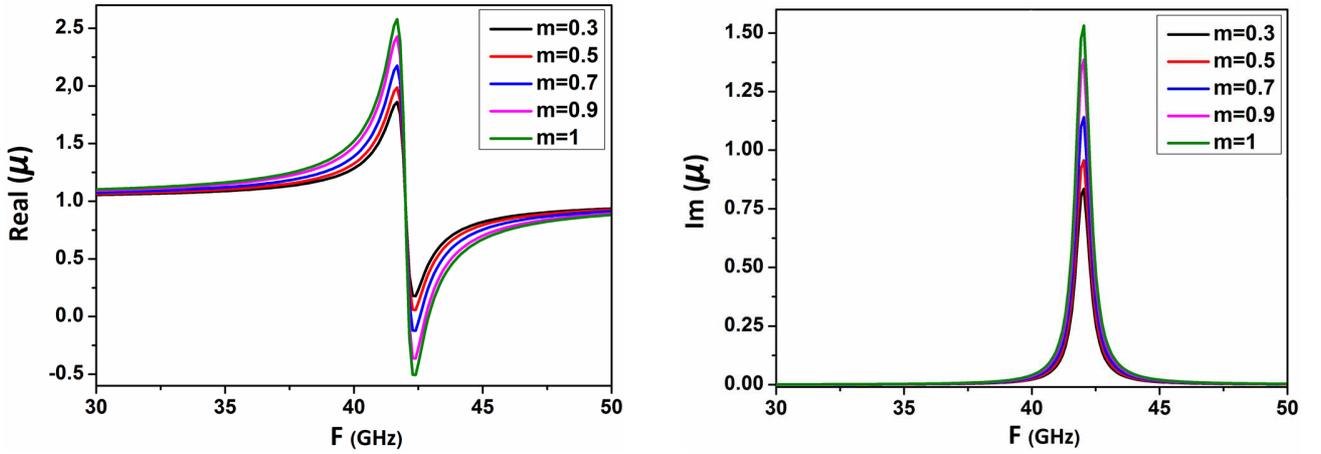


Fig. 6. Evolution of μ components as a function of CoFe_2O_4 nanocrystallites orientation.

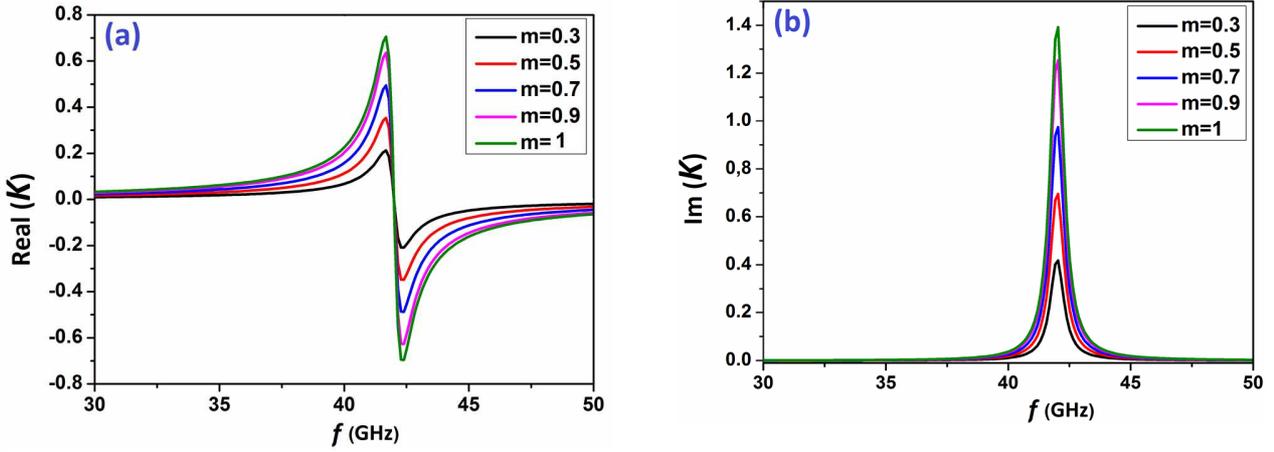


Fig. 7. Evolution of κ components as a function of CoFe_2O_4 nanocrystallites orientation.

As shown in the obtained results, by increasing the orientation of the nanocrystallites from 30 % to 100 %, the two elements μ and κ increase at the resonance while for the element μ_z the real part tends towards 1 and the imaginary part becomes zero for an orientation of 100 % magnetic material. As a result, the adopted model converges well towards the Polder model in the saturated case. The resonance frequency is practically unchanged.

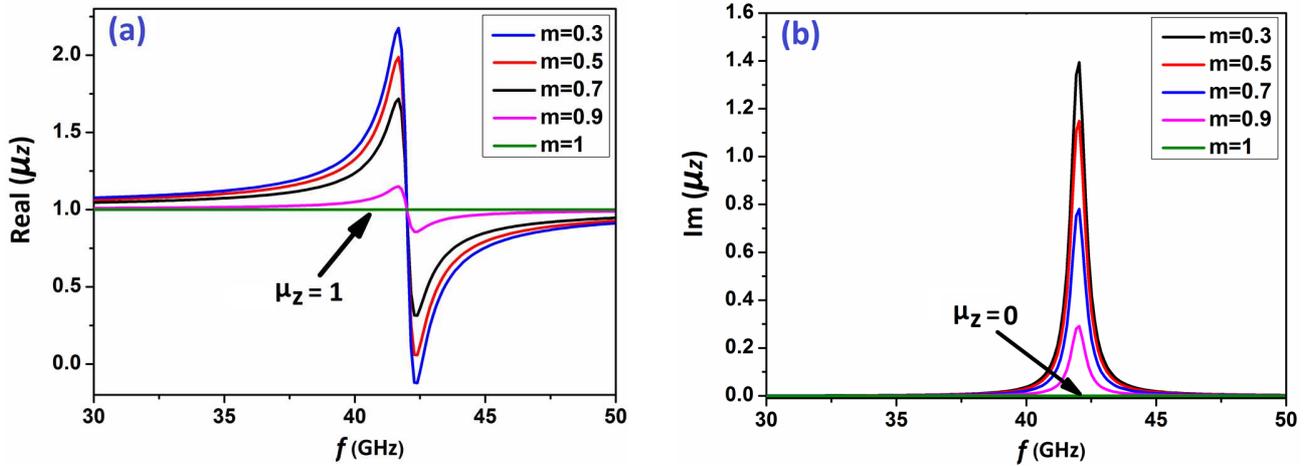


Fig. 8. Evolution of μ_z components as a function of CoFe_2O_4 nanocrystallites orientation.

3.2.2. Stripline circulator based on CoFe_2O_4 nano-composite

The Y-junction stripline circulator designed by Bosma is the most widely used in theoretical studies of circulators. As illustrated in Figure 8, the core of this circulator of stripline technology consists of three access lines oriented at 120° to each other and connected to a central metal disk [29]. This conductor is inserted

between two ferrite cylinders with the same shape as the central disk, in order to ensure the homogeneity of the transverse structure. The stripline structure is sandwiched by two ground planes above and below the ferrite disks. Two permanent magnets located on each side of the structure create a static magnetic polarization field that magnetizes the ferrimagnetic material along the axis of the magnetic cylinders.

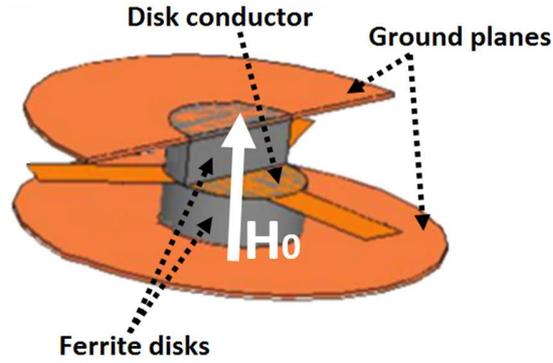


Fig. 9. Structure of the Y-junction stripline circulator.

As a first approach, we determined the effective permeability of the nanowired CoFe_2O_4 composite. In our study, we considered that we have CoFe_2O_4 nanowires oriented along the wire axis. Thus, the magnetic substrate has a saturation magnetization M_s of 0.18 T and an internal static magnetic field H_i of 1.52 T (it is the sample electrodeposited and cooled under a magnetic field of 0.6 T).

The evolution of the effective permeability of the CoFe_2O_4 nanowires as a function of frequency was modelled using the following formula:

$$\mu_{eff} = \frac{\mu^2 - \kappa^2}{\mu} \quad (5)$$

Here μ and κ are the calculated tensors by taking $m = 0.6$, which is the best squareness ratio value determined for our CoFe_2O_4 nano-composite.

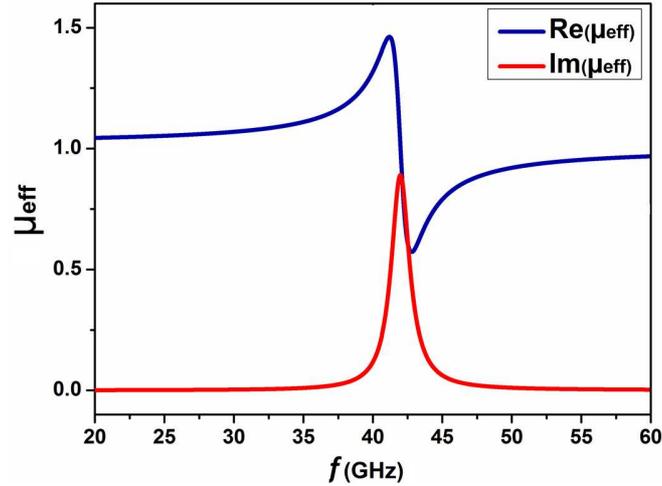


Fig. 10. Evolution of the effective permeability for CoFe_2O_4 nano-composite as a function of frequency.

As shown in Fig. 10, the resonance phenomenon of the effective permeability, when the internal field is fixed at 1.52 T, appears at a frequency of 42 GHz. Our circulator should operate around 40 GHz. However, this component does not operate at resonance (high losses); but either below or above resonance. The operation of the circulator known as above and below resonance are understood to be in field, i.e. when the internal field is respectively higher or lower than the field required for the resonance. The geometrical parameters of the central conductor of the circulator have been approximately determined using the Bosma's theory which depend on the effective permeability of the CoFe_2O_4 nanowires. The estimations provide a radius R of the central conductor disk of 475 μm , and an access line width W of 72 μm .

Once the effective ferrite permeability has been evaluated, the values of the S-parameters between two consecutive ports of the circulator, as a function of frequency, can be analytically determined from the theoretical analysis performed previously. The conductor material is the copper. The dielectric material is the alumina surrounding the ferrite disks, which has a relative permittivity of ($\epsilon_d = 10$). The permittivity value of the cobalt ferrite is around 13. A dielectric loss tangent ($\tan \delta = 10^{-2}$) has been taken into account in the analytical calculation. As can be shown in Figure 10, the modelled circulator using Matlab (Bosma) software operates at frequency of 35.8 GHz.

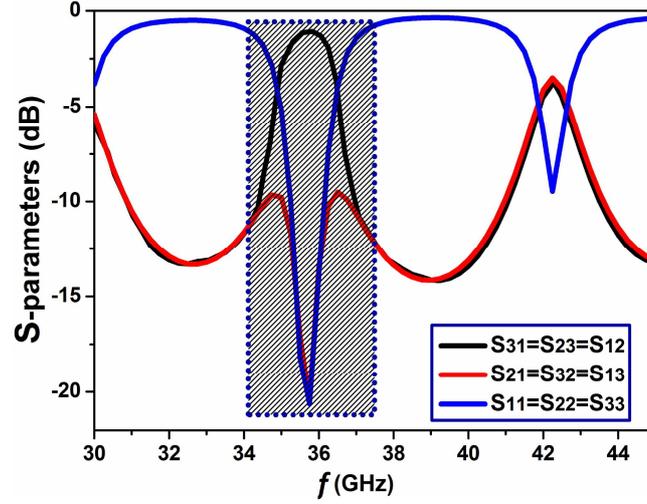


Fig. 11. *S-parameters magnitudes* for the stripline circulator; analytical calculation.

According to the evolution of the S parameters as a function of frequency, the reflection is 20.45 dB, the isolation is 20.64 dB and the insertion loss is 0.98 dB. This is the best result obtained with the parameters given previously.

As the structure concerned in our work is the coplanar topology, it does not correspond exactly to the one studied by Bosma, even though the operation principle remains the same. Therefore, the optimized geometrical parameters (R,W) will be maintained in the coplanar structure for the next analysis.

3.2.3. Integration of CoFe_2O_4 permeability tensor in HFSS

The anisotropic nature of the nanowires can be modeled using HFSS, either by using Polder formulations or by setting a user-defined permeability tensor. Conventionally, the Ansys HFSS uses the Polder model to calculate the components of the permeability tensor. However, the Polder model is only valid for the saturated state of the magnetic material [34].

When the applied field is along z direction, the permeability tensor can be expressed as,

$$\hat{\mu} = \begin{pmatrix} \mu' - j\mu'' & -j(\kappa' - j\kappa'') & 0 \\ +j(\kappa' - j\kappa'') & \mu' - j\mu'' & 0 \\ 0 & 0 & \mu'_z - j\mu''_z \end{pmatrix} \quad (6)$$

The last versions of this software allow to define the nine components of the permeability tensor as frequency-dependent functions. This makes it possible to integrate our adopted model for the permeability of the CoFe_2O_4 nano-composite into HFSS, in order to accurately predict the microwave response of the targeted component.

Our approach is based on the integration of the data points (calculated from the theoretical model in Matlab software) and on the proceeding of their linear interpolation into frequency dependent functions. The real and imaginary parts of the permeability tensor components are imported as separate datasets into HFSS. Each dataset is interpolated into frequency-dependent functions using an intrinsic piecewise linear interpolation function.

3.2.4. Simulation of a coplanar circulator based on CoFe_2O_4 nano-composite

The circulator uses coplanar topology with a bottom ground plane as shown in Fig. 12a. The coplanar structure, as the name suggests, is a topology having its conductor planes in the same level separated by a small distance G (Fig. 12b).

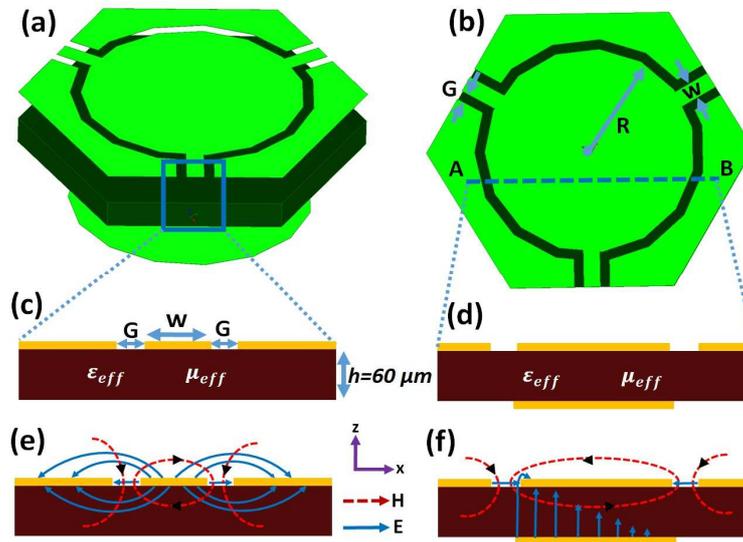


Fig. 12. Coplanar topology; (a) coplanar circulator with a bottom ground plane, (b) top view of the coplanar circulator, (c) cross sectional view of the CPW of the circulator, (d) A-B cross-sectional view of the circulator with ferrimagnetic substrate, (e) schematic distribution of the RF fields for a CPW line, (f) field displacement in the center of the circulator with coplanar junction.

It should be noted that the access line width W and the radius R of the central disk have been approximatively determined in the previous section. Moreover, the radius of the bottom ground plane disc is defined as the sum of the width of the central radius and the spacing gap G in order to improve the phenomenon of the electric field displacement at the central part of the circulator. So, keeping the value of W and R , the only remaining task is the optimization of G . To obtain the right G -value and to evaluate the performance of our ferrimagnetic nano-composite, simulations of the CPW, which ensure the input and output of the EM signal to the circulator, were carried out.

The nanowired ferrimagnetic substrate employed in our work is an alumina membrane, electromagnetically charged by CoFe_2O_4 nanocrystallites, that forming 60 μm in thickness. Before proceeding to the HFSS simulation, we have used a simple law of proportionality in order to approximate the effective permittivity ϵ_{eff} of the nano-composite, determined as a function of the nanocrystallites concentration and the AAO membrane pores filling coefficient [35]:

$$\epsilon_{eff} = (1 - p)\epsilon_{AAO} + \rho_{\text{CoFe}_2\text{O}_4} p \beta \epsilon_{\text{CoFe}_2\text{O}_4} \quad (7)$$

where p is the porosity of the AAO membrane, β the filling coefficient of the AAO membrane pores by CoFe_2O_4 nanocrystallites, $\rho_{\text{CoFe}_2\text{O}_4}$ the concentration of CoFe_2O_4 nanocrystallites contained in the pores, ϵ_{AAO} the permittivity of alumina, and $\epsilon_{\text{CoFe}_2\text{O}_4}$ the permittivity of bulky CoFe_2O_4 .

The typical characteristic properties of the ferrimagnetic nano-composite are listed in Table 1.

Table 1: Characteristics of CoFe_2O_4 nano-composite.

β (%)	p (%)	ϵ_{AAO}	ϵ_{eff}	$\mu_0 M_s$ (T)	ΔH (Oe)	$\tan \delta$	α	H_i (T)
100	50	10	8.2	0.18	284	10^{-2}	0.017	1.52

We must keep in mind that we used CoFe_2O_4 nanowires in order to benefit from its strong magnetocrystalline anisotropy to prevent the application of an external field. Subsequently, the excitation field of the component is limited in our case to the anisotropy field of the CoFe_2O_4 nanowires.

The coplanar circulator has been dimensioned according to the previously optimized values and then simulated on HFSS (Fig. 13). The circulator was modeled and adapted to have the input impedance of the ports equal to 50 Ω .

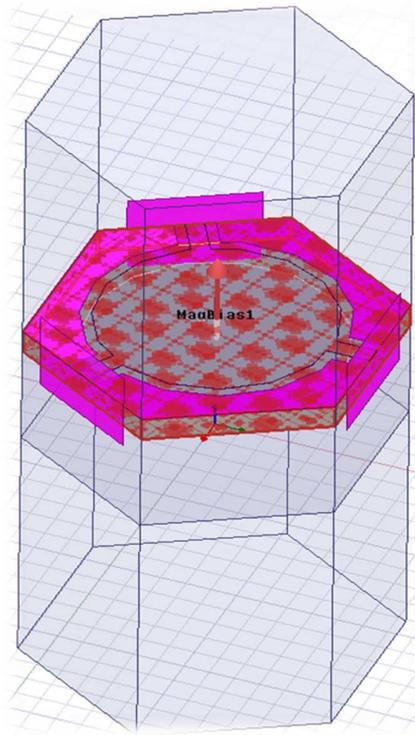


Fig. 13. Simulated coplanar circulator on HFSS.

Fig. 14 shows the simulation results of the designed circulator.

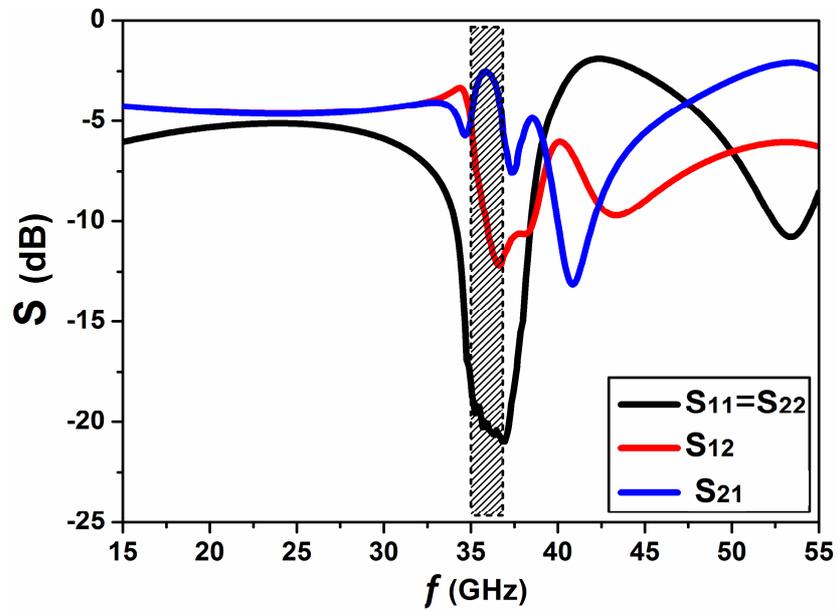


Fig. 14. *S parameters magnitudes* of the simulated coplanar circulator.

As shown in the simulation finding, one can observe a remarkable operation of the circulator around 35.7

GHz for a bandwidth of 1 GHz. In this band, the insertion losses are -2.57 dB, the insulation varies from 6 to 10.33 dB and the reflection coefficient is around -20.2 dB. A non-reciprocal effect of the circulator based on CoFe_2O_4 nano-composite can be observed. This requires the fabrication of the circulator as well as its microwave characterization.

3.2.5. Fabrication and microwave characterization of the coplanar circulator

After designing and dimensioning the coplanar circulator through various analytical and numerical studies, prototypes were fabricated and characterized. The fabrication process uses the different technological steps. The prototypes are then characterized using a microwave measurement bench consisting of a vector network analyzer (VNA) coupled with a probe tester. The measured S-parameters enable to conclude about the performance of the prototypes. No external magnetic field is applied during these experimental measurements. This makes it possible to check that the concept of self-biased circulator is achievable with our ferrimagnetic nano-composite.

3.2.5.1 Fabrication protocol of the circulator

The realization of the structures modeled under HFSS, requires many technological processes. Fig.15 illustrates the different steps required to fabricate a coplanar circulator based on the ferrimagnetic nano-composite.

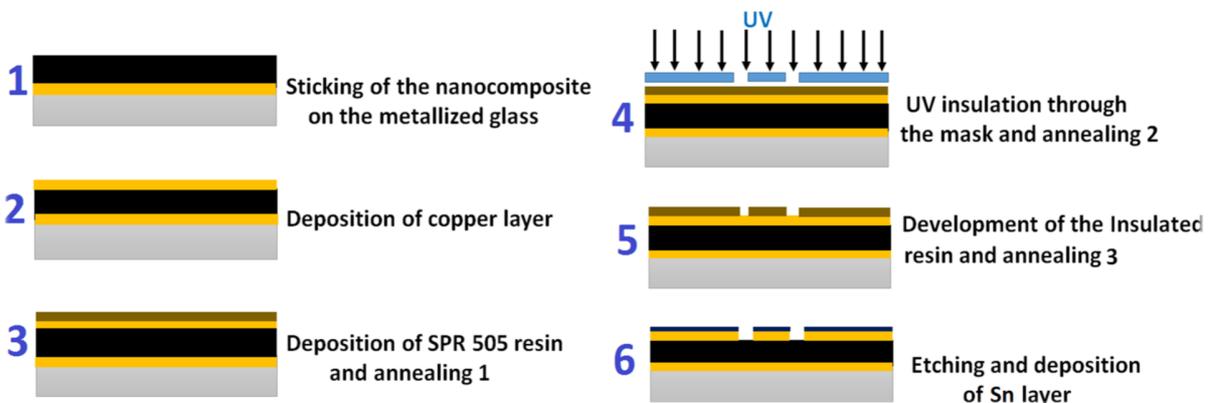


Fig. 15. Technological steps for the fabrication of the circulator in the clean room.

The preparation of the ferrimagnetic nano-composite involves the deposition of copper, the sticking and the grinding processes. A glass slide serves as a mechanical carrier for the ferrimagnetic nano-composite on which a $5 \mu\text{m}$ layer of copper is deposited. This first layer of copper acts as a bottom ground plane of the circulator. The copper is deposited by the TSD350 rf sputtering equipment. After, the ferrimagnetic nano-composite is bonded on the glass slide using the "Geo-fix" glue. Once glued, the ferrimagnetic substrate is polished with a very fine optical paper in order to obtain a roughness close to 100 nm which will favors a good adhesion of the copper that will be deposited to make the conductor and the top ground planes.

After the substrate cleaning with acetone and distilled water, the next step of the photolithography is initiated in order to transfer the coplanar pattern from the mask to the ferrimagnetic substrate. Then, a thin layer of SPR 505 resin is deposited via the spin coating device. When the resin deposition is finished, the substrate is heated at 90 °C for 90 s. This annealing step aims to evaporate quickly the solvents. The resin-coated substrate is placed in a UV contact exposure machine. The substrate-mask contact must be perfect to avoid any diffraction phenomena. It is insulated by the UV light for 3 seconds with a power of 270 Watts. To activate the polymerization of the resin, a second annealing is carried out at 110 °C for 90 s. The UV-exposed resin is dissolved in the MF-319 developer and reveals the non-insulated resin. Once revealed, the substrate is slowly rinsed with distilled water and carefully dried with nitrogen before proceeding to a final annealing at 110 °C for 4 min. This last heat treatment makes the resin mechanically more resistant. The resin development step is followed by the chemical etching phase which consists on removing, using the iron perchloride, the copper that has not been protected by the resin. For this purpose, the substrate is placed in a diluted iron perchloride solution until the copper unprotected by the resin is removed. After, the substrate is rinsed with the distilled water in order to stop the reaction of the iron perchloride and make the conductive lines appear. Finally, in order to protect the copper and the etched circuit from oxidation and corrosion, it is necessary to tin it. The tinning process is chemically done with a cold aqueous Tin (Sn) solution.

Fig. 16 shows multiple coplanar circulators printed on the nano-composite with the theoretically and numerically optimized dimensions.

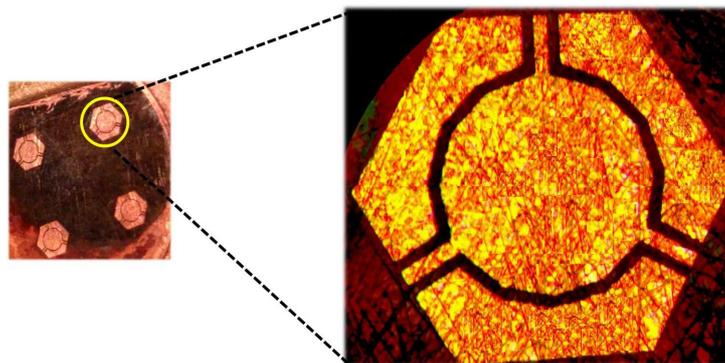


Fig. 16. Printed coplanar circulators on the nano-composite surface.

3.2.5.2. Microwave characterization

The microwave characterization has been performed to evaluate the performance of the fabricated prototypes. A two-ports vector network analyzer (VNA) operating in the 40 MHz – 67 GHz band and

equipped with three coplanar GSG (Ground-Signal-Ground) probes and its holders has been used. A microscope is also associated with the measurement bench for positioning the probes on the millimeter sized components as shown in Fig. 17.

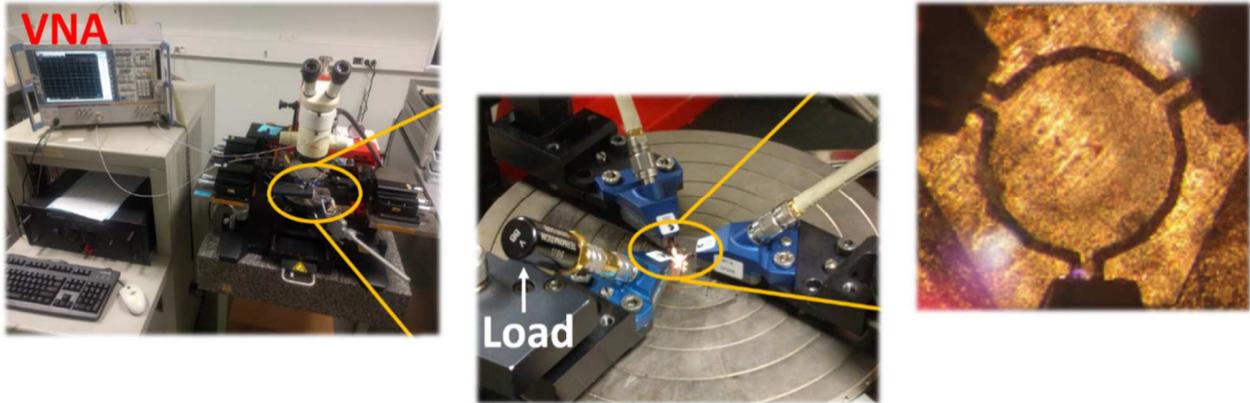


Fig. 17. Microwave characterization bench; measurement equipments and probes configuration.

Concerning the Y-junction circulator that has three access ports, it is necessary to have three points oriented at 120° . One of the third coplanar probes, used in the measurement, is connected to a load from 50Ω because even though the circulator is a 3-port component, it is possible to use only 2-port measurements with our VNA. It should be noted that in our measurements the resistance of 50Ω is sufficient to absorb practically the entire power of the incoming signal, so it can be qualitatively stated that the measured signal corresponds to the intrinsic response of the circulator. Before starting the measurements, an OSTL (Open-Short-Through-Load) calibration must first be performed for each port with a substrate suitable for the coplanar probes (calibration kit). This calibration will enable the analyzer to correct any systematic measurement errors, such as cable and connector errors. Afterwards, we can measure the S_{ij} parameters to visualize the circulation phenomenon for the coplanar circulator. To check the repeatability during the microwave measurement, the Load was connected to every port. The same microwave response was observed, this enables to draw a clear conclusion about the presence of the non-reciprocity phenomenon.

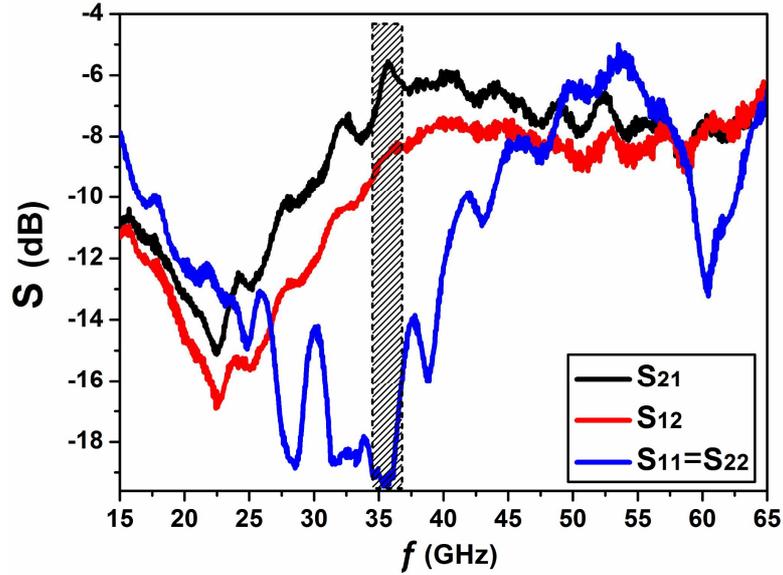


Fig. 18. *Measured S parameters magnitudes of the fabricated coplanar circulator.*

Fig. 18 represents the S parameters, measured on a frequency band from 15 to 65 GHz of the coplanar circulator made of the ferrimagnetic substrate, which has been separated into two components: reflection and transmission parameters. It should be noted that no magnetic field was applied during this phase. As shown in Fig. 18, the reflection curves at port 1 (S_{11}) and port 2 (S_{22}) show a good matching in the measurement frequency band.

Moreover, the ferrimagnetic nano-composite confers to the circulator its non-reciprocity. The reflection level is -19.3 dB at 35 GHz. The transmission levels are -5.57 and -8.59 dB for the insertion (S_{21}) and isolation (S_{12}) loss, respectively. **Furthermore, it is observed that the non-reciprocal phenomenon is present almost over the range of 25-45 GHz, but the frequency that shows the lowest reflection is 35 GHz as predicted by HFSS. This information can be exploited as an additional tool to design multi-band circulators. For the present fabricated prototype.** The non-reciprocal phenomenon is about 3 dB and the circulation phenomenon is observable around 35.5 GHz. The observed non-reciprocal effect ($S_{12} \neq S_{21}$) appears to be small but is a first indication of the possible circulation function.

Although this first encouraging result, the high losses (5.57 dB) are mainly due metallic losses according to the low substrate thickness, and ferrite nanowires exhibiting more losses in the non-saturated state and worked near the gyromagnetic resonance. The magnetic state of the CoFe_2O_4 nanowire are certainly multi-domain. Owing to its atomic structure, the cobalt ferrite has an easy plane magneto-crystalline anisotropy, and the effective anisotropy of the magnetic composite substrate is rather a shape anisotropy according to the

nanowire shape.

In this regard, it can be generally pointed out that there is a difference between the simulation and the experimental results for the S-parameters, but at the resonance frequency (35 GHz) there is a close agreement between HFSS predictions and VNA measurements. The observed difference for the S-parameters could be mainly explained by the incomplete Irashagi model used in this work, since our nano-composite is partially magnetized. This model considers the geometrical parameters of the nanowires and the magnetization ratio of the ferrimagnetic nano-composite. A magneto-dynamic model, that will take into account the shape, magnetocrystalline, and surface anisotropies and the dipolar, spin-orbit interactions, will be required in a future work in order to accurately simulate the microwave response of such coplanar topology of the circulator [17].

From this first analysis and experimental study on circulator based on ferrimagnetic nanowires, other studies are under consideration to improve the nano-composite properties. Using thicker alumina membranes could facilitate all the fabrication steps in the clean room (annealing, cooper deposition, surface etching, etc). Moreover, the increase in thickness will increase the quantity of cobalt ferrite crystallites, which will be a bonus to improve the magnetic properties responsible for the enhancement of the microwave response.

Conclusion

In this study, we have electromagnetically embedded CoFe_2O_4 nanowires that exhibit a strong internal magnetic field. This nanowired substrate is selected for the fabrication of a microwave circulator. First, we have presented the theoretical model used to determine the permeability tensor of the ferrimagnetic nano-composite. The general functioning and analytical modeling of the circulator in a stripline topology was demonstrated. This modeling leads to a preliminary rapid evaluation of the component performance and its dimensioning before switching to the coplanar topology. To complete the dimensioning study of the coplanar circulator, several simulation tasks were carried out after the integration of the permeability tensor of the nano-composite on HFSS software. Moreover, a characterization of the prototype component was performed using a vector network analyzer. A non-reciprocal effect of 3 dB was observed for the constructed coplanar circulator which demonstrates the feasibility of achieving a non-reciprocal effect using our ferrimagnetic substrate. Moreover, our realized experiments in the synthesis of nano-composite and fabrication in a clean room have enabled us to identify the technological challenges and foresee new possibilities to develop magnetic nano-composites applied to the design of self-biased circulator.

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