



# JORNADA DOCTORAL DE JÓVENES INVESTIGADORES EN MAGNETISMO

17 OCTUBRE 2019

## *BOOK OF ABSTRACTS*

THE SPANISH MAGNETISM CLUB AND THE SPANISH CHAPTER OF THE  
IEEE MAGNETICS SOCIETY ANNUAL JOINT MEETING



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# Development of 3D advanced magnetic nanostructures fabricated by focused electron beam induced deposition

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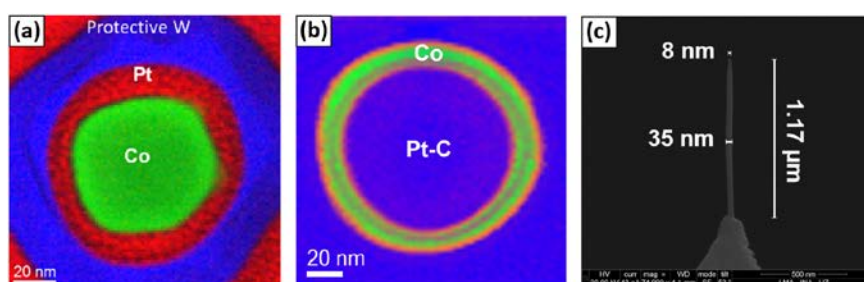
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The fabrication of three-dimensional (3D) magnetic nanostructures is currently a central topic in nanomagnetism [1]. Ferromagnetic nanowires (NWs) and nanotubes (NTs) are potential candidates for magnetic data storage, logic and sensing, and Focused Electron Beam Induced Deposition (FEBID) could play a crucial role in the fabrication of these architectures [2].

A new approach to grow core-shell heterostructures by FEBID has been developed [3], obtaining Co@Pt and Fe@Pt NWs, and Co NTs, as shown in Figure (a-b). In the NWs case, this strategy aims to minimize the degradation of the magnetic properties caused by the surface oxidation of the core to a non-ferromagnetic material. In addition, *ex situ* and *in situ* annealing treatments of Co and Fe NWs have been found to increase metal purity, crystallinity and magnetic induction [4,5,6].

Furthermore, we report on the fabrication of Magnetic Force Microscopy (MFM) tips by FEBID, showing great performance. Tailored Co and Fe MFM tips have been tested, both in ambient conditions and liquid environment, getting outstanding mechanical stability, resolution and sensitivity [7]. In particular, Fe tips with a ~7-8 nm-wide sharp end (Figure (c)) exhibit low sample-tip magnetic interaction, which has been useful to measure complex magnetic states such as skyrmions.



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*Index Terms* — Magnetic nanowires, nanotubes, focused electron beam induced deposition.

# Study of the Detection of a Single Magnetotactic Bacterium

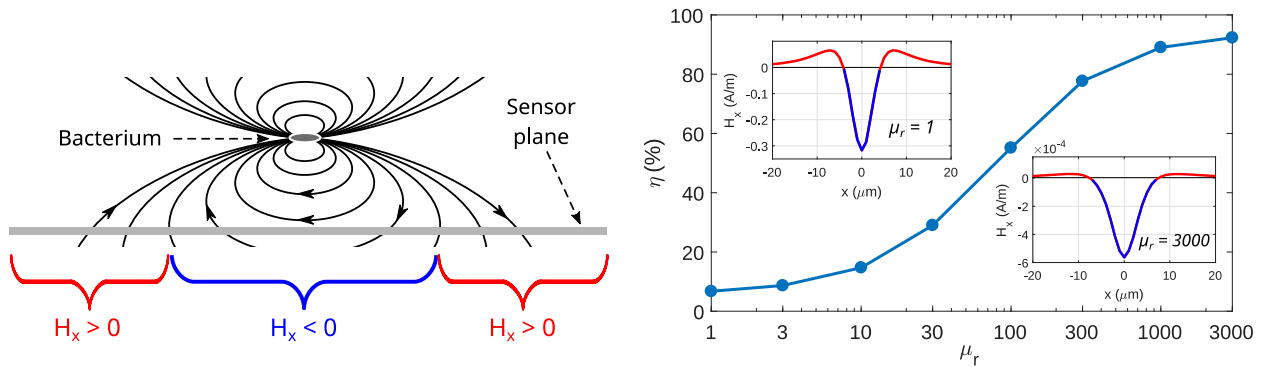
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Magnetotactic bacteria are aqueous microorganisms that navigate in the Earth magnetic field using their internal chain of magnetic nanoparticles, which actuate as a compass needle. For instance, the species *Magnetospirillum gryphiswaldense* contains up to 25 cuboctahedral particles of magnetite ( $\text{Fe}_3\text{O}_4$ ) with a size of about 45 nm. They are of great interest for biomedical applications, for instance as living micro-robots guided magnetically.<sup>1</sup> Aiming to detect the presence and movement of a single bacterium using a magnetic sensor, we study the characteristics of its field in the sensor position. Being much larger than the bacterium, the sensor is affected in different regions by fields with opposite sign. If the permeability of the sensor is low ( $\mu_r \sim 1$ ), the net effect of the positive and negative fields is almost fully cancelled, whereas with  $\mu_r \gg 1$ , the field distribution changes and the sensor output is greatly increased. We combine analytical and numerical calculations by finite elements to evaluate the performance of the sensor, as a function of the permeability of the material, for different geometries and sensing conditions.



Left: Horizontal component  $H_x$  of the bacterium field in the sensor position. Right: Dependence, with the permeability of the sensor, of the figure of merit  $\eta$ , based on the double integral of  $H_x$  over the sensor area.  $\eta$  takes values close to 0 when the permeability is low, because the contribution of positive and negative fields regions nearly cancel each other. When the permeability is high,  $\eta$  approaches 100 % as one of the regions dominates.

*Index Terms* — Magnetotactic bacteria, permeability, magnetic sensor, finite element method.

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# Role of the defects on the magnetic properties in the shape memory alloys

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Metamagnetic shape memory alloys are being widely studied during the last decade because of the unique multifunctional features they show as a result of the interplay between a martensitic transformation and a complex magnetic ordering. The induction of the structural transformation by an applied magnetic field may give rise to interesting properties such as the magnetic shape memory effect, large magnetoresistance or giant inverse magnetocaloric effect, that make these alloys very attractive for practical applications such as sensing and magnetic refrigeration. Reduction in the size of particles may optimize the magnetocaloric effect, either by increasing the temperature range or by reducing the hysteretic losses. On the other hand, the integration of powder alloys into a polymer to form composites has been found to be especially interesting for the development of magnetically-controlled dampers and actuators.

In the present work, the influence of mechanically-induced defects on the magnetostructural properties has been analysed in a Ni-Mn-In-Co alloy subjected to mechanical milling. Even for soft hand-crushing, a large amount of microstructural defects are induced on the alloy. These defects, which cause a large stabilization of the martensite, degrade the martensitic transformation characteristics and the magnetic response of the alloy. This behavior is compatible with the inclusion of non-transforming antiferromagnetic regions in the alloy as a result of milling. In particular, it is observed that the magnetocaloric effect value decreases with the increasing amount of defects. Nevertheless, even though the MCE value in the powder is lower than in the bulk, the broader temperature range for the martensitic transformation in the powder can make the Refrigeration Cooling Power be comparable to that in the bulk. Therefore, the obtained microparticles can be considered as good candidates for magnetic refrigeration applications at the microscale.

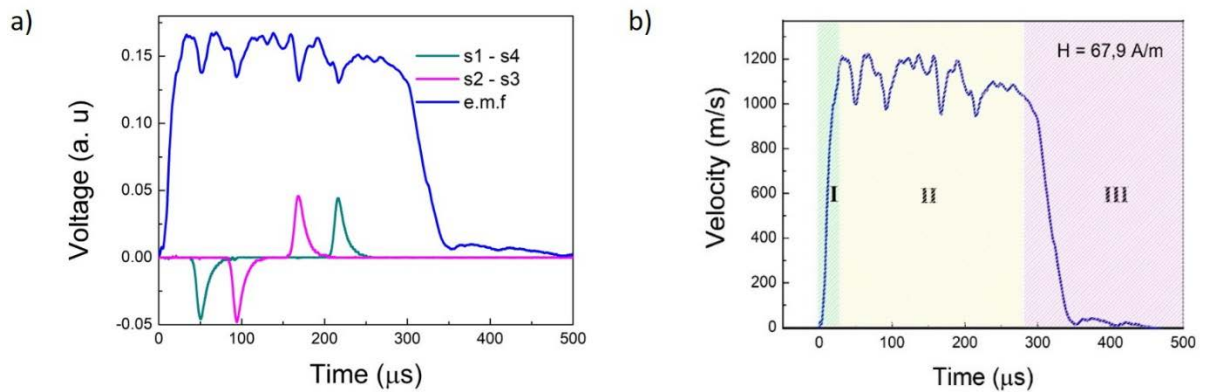
*Keywords* — Magnetic shape memory alloy, mechanical milling, defects, magnetocaloric effect.

# Time-resolved velocity of a domain wall in a magnetic microwire

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The dynamic process of nucleation, propagation and braking of a single domain wall (DW) has been systematically determined in a Fe-based magnetostrictive microwire under the action of an axial magnetic field. While in previous reports the Sixtus-Tonks experiments have provided partial information on the process (i.e., the average velocity), in the present study we report on the instantaneous processes involved in the propagation of the DW, as well as the transient process during the DW depinning. The experimental measurements were carried out using the spontaneous Matteucci effect induced during DW propagation due to the small helical magnetization component created during the fabrication process.



a) Voltages induced in the pairs of Sixtus-Tonks pickup coils s1-s4 and s2-s3, and electromotive force induced at the ends of the microwire during the depinning and propagation of a single domain wall. b) Time-resolved domain wall velocity: three main regions can be distinguished and associated to the acceleration of the DW (I), propagation (II) and final braking when the DW goes out of the primary coil (III).

*Index Terms* — Magnetic microwire, domain wall motion, time-resolved domain wall velocity.



# FORC study of magnetic phases in feldspars and ofites.

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FORC study in feldspars [1] showed that the magnetic separation process of this material is selective because of the existence of uncoupled hard (goethite) and soft (magnetite) phases. The soft phase has higher magnetic moment than the harder one. To validate our explanation for the selectivity of the separation process, we studied ofites, which are rocks of subvolcanic holocrystalline nature from magmas that did not reach the surface completely, solidifying at a shallow depth. Ofites have a great industrial importance since they are used as a basis of stability helping the distribution of pressures, vibrations and drainage of the railways as well as in the asphalt layers of roads and highways. Measurements of hysteresis loops and FORC distributions, in combination with X-ray diffraction, show the presence of magnetite and hematite. The magnetic separation process is not selective, which can be explained by the absence of phases with very different magnetic character. This is in agreement with the interpretation of the FORC results of the magnetically separated feldspar samples.

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# High magnetization FeCo nanoparticles synthesized by chemical and physical routes

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FeCo binary alloys have excellent soft magnetic properties as high magnetization saturation and large permeability. As nanoparticles (NPs) they can be used as magnetic fillers for new formulations of magneto-rheological fluids [1]. Here we present results concerning the synthesis and characterization of FeCo and FeCo–V high magnetization NPs fabricated by chemical (reduction or thermal decomposition from metallo-organic precursors) and physical (laser ablation in liquid and ball milling) routes. The particles have been characterized by Dynamic Light Scattering (DLS), X-ray diffraction (XRD), Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) to determine the size, structure, morphology, and by Vibrating Sample Magnetometry (VSM) to obtain magnetic parameters. Chemical reduction of Fe(III) and Co(II) salts in the presence of  $\text{AlNH}_4\text{F}_4$  yields 20-50 nm laminar and acicular particles of FeCo NPs with  $M_s$  about 210 emu/g. Thermal decomposition leads spherical NPs but with lower  $M_s$  values (115 emu/g). To test physical routes, the starting material has been Vacoflux 50<sup>®</sup> ( $\text{Fe}_{49}\text{Co}_{49}\text{V}_2$ ) from Vacuumschmelze. A picosecond pulsed laser at  $\lambda=355$  and 532 nm in acetone produced NPs about 10-50 nm with saturation magnetization of 7  $\mu\text{emu/ml}$ . By High Energy Ball milling we have obtained 0.5-1  $\mu\text{m}$  NPs with *bcc* structure and about 195 emu/g saturation magnetization. Either route provides good quality NPs. The main differences among them concern the final quantity and morphology of the obtained products.

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*Index Terms* — Iron-cobalt nanoparticles, saturation magnetization, chemical reduction, thermal decomposition, laser ablation, mechanical ball milling.

# Spinel Ferrite Nanoparticles for Magnetic Lateral Flow Immunoassays

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Lateral Flow Immunoassay (LFIA) is a paper-based architecture whose most popular use is the pregnancy test. They are increasingly being used for determination of biomarkers, allergenic pathogens, drugs and metabolites, biomedical, food safety and environmental settings [1]. Their sensitivity, selectivity, quickness and ease of use make them ideal for Point-of-Use (PoU) testing. One of the key points of the LFIA is the labelling of the biomarker, traditionally with latex or gold nanoparticles, which provides a visible signal. These are essentially qualitative (presence/absence) or semi-quantitative analyses. To add quantification capacities to LFIA, the use of Magnetic Nanoparticles (NPs) has been proposed [2].

The magnetic LFIA has to be associated to a magnetic reader that should be itself fast and portable. A radio-frequency inductive sensor has been developed for this purpose which takes advantage of the superparamagnetic character of the NPs [3,4]. The purpose of this work was to carry out a systematic study of the effect of different nanoparticles in order to provide insights on the sensing principle. Superparamagnetic metal oxide NPs of spinel ferrites (SFs), with a general formula  $M^{2+}Fe_2^{3+}O_4$  where M can be a divalent metal such as Co, Mn, Ni, Zn, etc., are suitable for this study as a result of their multifunctional properties, affordability and fine-tuning capability of their properties by simple chemical manipulations [5]. Three different composition SFs nanoparticles ( $Fe_3O_4$ ,  $Ni_{0.31}Fe_{2.69}O_4$  and  $Mn_{0.13}Fe_{2.87}O_4$ ) have been characterised to find out the optimum properties for LFIA. Particle size, crystallinity and magnetic properties such as initial permeability and saturation magnetisation were studied and compared. The results indicate that magnetite SFs, which have the highest initial permeability, are the most suitable labels yielding the highest quantitative signal.

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# Soft magnetic composite filaments with enhanced uniformity for additive manufacturing

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Additive manufacturing (AM), which builds objects through the layer-by-layer deposition of material, is a design revolution for today's construction of 3D objects. Among the different AM techniques, fused deposition modeling (FDM) is one of most widely used methods. 3D printers based on FDM print objects from a filament, usually a thermoplastic, that is fused into the head of the printer to be deposited by a nozzle.

Nowadays, there is a growing interest to extend AM to functional applications and that will require the use of composite filaments with functional properties for 3D printing. In the case of soft magnetic composite filaments, there is a large restriction for research purposes due to the lack of variety in the commercially available products (only one type is in the market) while there is no report in the literature showing laboratory fabrication of such filaments. In addition, homogeneous composite filaments are essential to fabricate parts with predictable and uniform characteristics especially if they will be used for applications, such as flux concentrators or transformer cores. Furthermore, the typical way of composite filament fabrication requires the mixing of particles with the polymer pellets in a kneader, which is usually industrially available. On the other hand, alternative mixing available for performing in laboratories can lead to loss/agglomeration of magnetic powder in the hopper or a non-uniform ratio of the polymer and the magnetic particles progressing through the mixing zones in the extruder. Both of these will cause problems with unpredictable amount of particle loss, repeatability and inhomogeneities of the final filament.

In this work, an original procedure for fabricating composite filaments for FDM was designed and developed. Customized polymer capsules were fabricated and filled with soft magnetic maraging steel as the feedstock for the filament extrusion. Results from both microstructural (X-ray tomography and scanning electron microscopy) and magnetic characterization reveal a good agreement between the actual and nominal compositions and a uniform particle distribution within the composite filament. This method, not limited to magnetic particles or any specific polymer, does not require sophisticated machinery yet allows desired compositions with good control of the amount of particles added.

*Index Terms* — Magnetic composite filaments, additive manufacturing, uniform particle distribution.

# Synthesis of iron oxide nanoparticles with tunable morphology and composition

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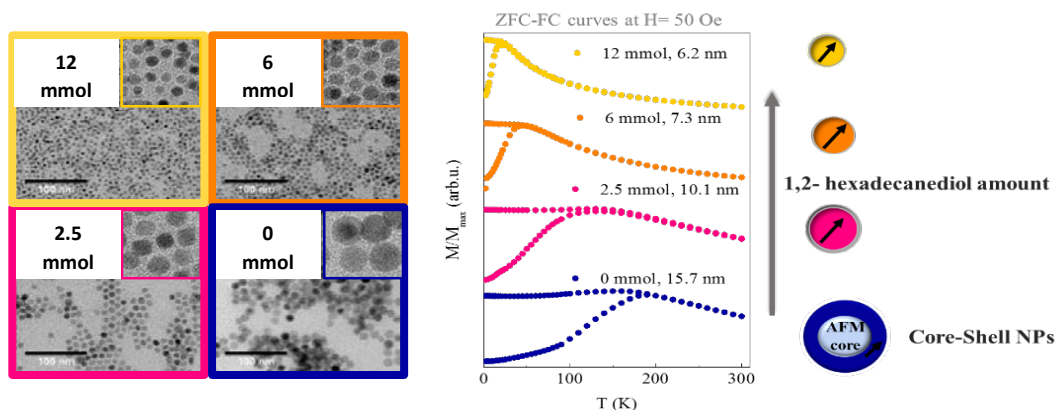
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Iron oxide nanoparticles (NPs) with  $\text{Fe}_{3-x}\text{O}_4$  composition have attracted great attention in biomedical proposes because of their good biocompatibility and magnetic performance. The synthesis method has a strong effect on structural and magnetic properties of the particles and the thermal decomposition method allows to tune these properties depending on the synthesis conditions<sup>1,2</sup>.

The overall aim of this work is to improve the reproducibility and to optimize the synthesis in order to tune each sample to its specific application. Our results show the key role of the concentration of the 1,2-hexadecanediol and the solvent 1-octadecene during the reaction. We observed that greater amounts of either compounds inhibit the  $\text{Fe}_3\text{O}_{4-x}$  NP growth and diminish the reaction yield, whereas small amounts increase the reaction yield and induce the formation of parasitic phases such as wüstite. Because of their structural and compositional variability, the samples exhibit two distinct magnetic behaviors at low temperatures. Smaller NPs show a single peak in the zero-field cooling-field cooling (ZFC-FC) curves below 200 K. In contrast, the bigger ones display two peaks in the ZFC-FC curves and an exchange bias in the hysteresis loops at 5 K after field cooling at 1 T. We associate these phenomena with the interaction between the ferrimagnetic (magnetite) and antiferromagnetic (wüstite) phases.

The work was supported by Spanish MINECO (MAT2015-68772-P; PGC2018-097789-B-I00) and European Union FEDER funds. M.ET. and T.G acknowledge Spanish MINECO for a Ph.D. contract (BES-2016-077527) and Erasmus+ program for a research internship, respectively.

*Index Terms* —iron oxide nanoparticles, synthesis optimization, structural and composition inhomogeneities



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# Quantitative analysis of the longitudinal spin Seebeck Effect

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Spin Seebeck effect (SSE) is defined as the excitation of a spin current in a magnetically ordered material (FM) by its subjection to a thermal gradient [1]. This spin current has been proved to be carried by collective magnetic excitations —magnons— rather than mobile carriers [2], and it is usually detected by its conversion into an electrical current by means of the Inverse Spin Hall effect in an attached metallic and non-magnetic material (NM), typically Pt. Thus, SSE is a complex phenomenon whose output is the result of the interplay between several parameters characterizing magnetic, electrical, magnonic, and thermal properties of the involved materials and interfaces, making its quantitative analysis far from straightforward. In addition, two different physical mechanisms contribute to the spin current in SSE: the interfacial temperature difference, and the bulk magnon accumulation in the magnetic thin film due to the thermal gradient along it.

Here we report a series of experiments and calculations intended to enable a quantitative analysis of the longitudinal SSE (LSSE) in bilayers of epitaxial thin films of ferrimagnetic insulating maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ , [4]) as FM, and Pt as NM. The use of an insulating FM overcomes theoretical and experimental complications, and brings some advantages for practical applications.

We study the temperature dependence of the stationary LSSE, and correlate it with the thermal conductivity of both the magnetic thin-film and the substrate, measured by the  $3\omega$  method [3]. The influence of the magnetic layer thickness is also determined and considered in the analysis. As a result, we obtain the value of different parameters that influence the spin transport and, in addition, the contribution of each of both mechanisms can be weighted.

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*Index Terms* — thermal spintronics, Spin Seebeck effect, thin film thermal conductivity, ferromagnetic insulators.

# Walker-like domain wall breakdown in layered antiferromagnets driven by staggered spin-orbit fields

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The magnetization switching process is related to the propagation of a magnetization reversal mode. Ultrafast propagating modes are expected from theory in antiferromagnets (AFMs), since the maximum speed of moving domain walls (DWs) can be up to two orders of magnitude higher than in the case of ferromagnets (FMs). Moreover, the steady-state motion of magnetic textures in FMs is limited by the so-called Walker breakdown (WB) [1], which is characterized by intrinsic instabilities of the DW structure, which translates into slower dynamics. Additionally, moving antiferromagnetic textures obeys the precepts of special relativity, meaning that the DW speed is superiorly bounded by the maximum group velocity of the magnons of the medium,  $v_g$ , and that it contracts as it approaches  $v_g$  [2].

Recently, it has been theoretically proposed [3] and experimentally demonstrated [4] how to excite dynamics in AFMs that present crystalline lattices in which the magnetic atoms have a local environment with broken inversion symmetry and where the two magnetic sublattices form inversion partners, as in  $\text{Mn}_2\text{Au}$  and  $\text{CuMnAs}$ . This is possible through the phenomenon known as Néel spin-orbit torque, in which a staggered spin-orbit field is generated at the sublattices of the system through the injection of an electric current in each of the ferromagnetic planes.

In this talk, we present a study on the dynamics of DWs in a  $\text{Mn}_2\text{Au}$  system driven by a staggered spin-orbit field using atomistic simulations and analytical theory. We observed that when a DW is driven at high spin-orbit fields a nucleation process of magnetic textures and the appearance of a supermagnonic dynamical regime for some of the DWs of the system take place. As a consequence of the speeds reached in the aforementioned supermagnonic regime, spin waves (SWs) are emitted as a way of dissipating energy, which is known as the spin Cherenkov effect [5], which takes place when the minimum phase velocity of the SWs of the medium is exceeded.

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*Index Terms* — dynamics of domain walls, antiferromagnets, Walker breakdown, supermagnonic regime, nucleation of magnetic textures, spin Cherenkov effect.

# Evolution of structural and magnetic properties during mechanical alloying of Fe<sub>70</sub>Zr<sub>30</sub>

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Mechanical alloying has a unique capability of producing thermodynamically disfavored reactions and destabilizing equilibrium systems [1]. However, several aspects inherent to powder samples affect magnetic properties of samples produced by this technique. For example, in the case of the magnetocaloric effect, a general decrease of the peak value ascribed to the Curie transition of the amorphous phase and a large broadening of the thermal dependence of the magnetic entropy change are observed due to the presence of inhomogeneities in the amorphous matrix [2]. These inhomogeneities lead to the existence of a non-negligible distribution of Curie temperatures [3], whose corresponding parameters (average value of the Curie temperature,  $\overline{T_C}$ , and its standard deviation,  $\Delta T_C$ ) can be obtained from the analysis of the approach to saturation curves [4].

In this work, microstructure and magnetic properties of a mixture of pure 70 at. % Fe and 30 at. % Zr produced by mechanical milling have been characterized as a function of the milling time. Microstructure and Fe environments results show the formation of an almost fully alloy after 50 h of milling. The enhancement in the soft magnetic behavior of the samples with the increase of milling time is ascribed to the averaging out of the magnetocrystalline anisotropy as crystal size decreases and ferromagnetic amorphous fraction increases. However, the magnetoelastic large order anisotropy (in the range of the powder particle size) prevents a decrease of coercivity,  $H_C$  below 1 kA/m. The obtained exponent,  $n$ , describing the magnetic entropy change ( $|\Delta S_M| = aH^n$ ) at Curie temperature for a pure amorphous phase is higher than the expected values for amorphous alloys [5], which is ascribed to the compositional inhomogeneity of the samples due to the the remnant nanocrystalline phase. The decrease of  $\Delta T_C$  with the milling time is in agreement with the microstructural results, showing the depletion of the  $\alpha$ -Fe phase inside the amorphous matrix and the increase of the amorphous fraction, responsible of the studied magnetic transition.

**Keywords:** mechanical alloying and milling, amorphous and nanocrystalline materials, magnetic entropy change, Mössbauer spectroscopy, soft magnetic materials

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# Large amplitude, nanoripple array of soft magnetic film grown on pre-patterned polymer foils: surface morphology and uniaxial magnetic anisotropy

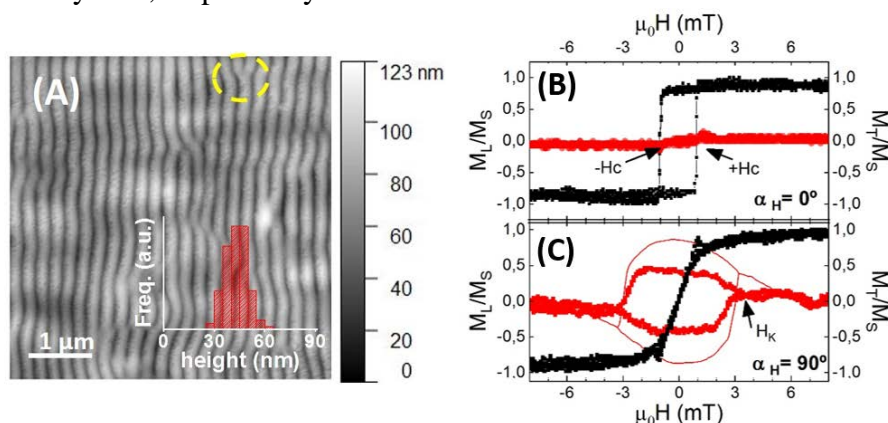
Elena H. Sánchez<sup>1,2</sup>, Rodrigo Aragón<sup>1</sup>, Miguel A. Arranz<sup>2</sup>, Gabriel Rodríguez-Rodríguez<sup>1</sup>, Esther Rebolgar<sup>3</sup>, Marta Castillejo<sup>3</sup> and Jose M. Colino<sup>1</sup>

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High performance novel magnetic materials require the study and tuning of induced anisotropy and reversal fields in order to develop new functionalities in extended magnetic media. To this purpose we have explored the nano-undulated soft magnetic films which result from the normal incidence, high vacuum deposition of permalloy ( $\text{Ni}_{80}\text{Fe}_{20}$ ) on a pre-patterned and nano-undulated surface of flexible and transparent polymeric foil of polyethylene terephthalate (PET). This substrate was previously processed by pulsed laser irradiation, achieving a linear and periodic ripple pattern, e.g., a laser-induced periodic surface structure (LIPSS), with a periodicity around 250 nm and average amplitude up to 58 nm. The nearly sinusoidal profile, linear pattern is of remarkably large amplitude, yet larger than the thickness of the continuous film grown on top of it. The dimensions of this undulated uniform magnetic media were studied by Atomic Force Microscopy. Vectorial MOKE (reflection) as well as Voight (transmission) effects were used to measure magnetization and critical fields at different in-plane field angles. Comparison of those with Stoner-Wohlfarth and other models confirms the induced uniaxial shape anisotropy along the rippled pattern, provides a measure of anisotropy strength  $\mu_0 H_K$  (3 – 9 mT) and points to different mechanisms for magnetization reversal: rotation or domain wall pinning, as the field is applied along the hard or the easy axis, respectively.



**Figure 1.** (A)  $5 \times 5 \mu\text{m}^2$  AFM topography image of the surface pattern of 20 nm thick permalloy film grown on a nano-undulated PET foil. The inset shows a ripple amplitude histogram from the surface pattern. Yellow circle surrounds an exemplary Y-shape defect or pattern dislocation. (B)-(C) Normalized longitudinal (black symbols) and transversal (red symbols) magnetization loops obtained from the longitudinal MOKE of the 15 nm thick permalloy film for the field applied along the easy-magnetic axis (B) and hard-magnetic axis (C). Red solid line is a loop calculated for such direction with Stoner-Wohlfarth model.

# Magnetic and photocatalytic properties of cobalt-zinc ferrites

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Spinel nanoferrites ( $MFe_2O_4$ ) with different metallic cations ( $M$ ) have been extensively studied in different technological fields as magnetic recording, photocatalysis, microwave absorption and drug delivery. The main objective of this work is to synthesize multifunctional nanoferrites with photocatalytic properties under visible light (energy band gap lower than 3.1 eV). Furthermore, their ferrimagnetic behavior allows separation and recycling of the catalyst under the action of an external magnetic field. While, Co-Zn nanoferrites have been widely investigated regarding the magnetic properties [1,2], few reports comparatively analyze both magnetic and photocatalytic response.

In the present work,  $Co_xZn_{1-x}Fe_2O_4$  ( $0 \leq x \leq 1$ ) nanoferrites were synthesized by co-precipitation method mixing stoichiometrically  $Fe(NO_3)_3 \cdot 9H_2O$ ,  $Zn(NO_2)_3 \cdot 6H_2O$  and  $Co(NO_2)_3 \cdot 6H_2O$  and controlling the pH with a NaOH solution (1 M), which was added until the solution reaches pH = 13 favoring the precipitation and complete hydrolyzation of the ions. The precipitates were centrifuged, washed with distilled water and dried at 50 °C in an oven. Finally, the samples were annealed in air at 400 °C during 6 hours [3].

The structure of samples was analyzed by X-ray diffraction (Siemens Diffractometer D5000) and TEM (Tecnai Field Emission Gun) and the magnetic measurements were carried out with a SQUID magnetometer (Quantum Design MPMS XL7). After sample characterization, their photodegradation activity was evaluated by measuring the degradation of phenol in aqueous solution under visible light with a Xe arc lamp with a 400 nm cutoff filter. The results show maximum values of the magnetic moment for  $x = 0.7$  as a consequence of the cation distribution in the spinel structure. Regarding the photocatalytic activity, maximum phenol degradation ratios under visible radiation are found for  $x = 0.5$ . Thus,  $Co_{0.5}Zn_{0.5}Fe_2O_4$  nanoferrite displays optimum response in terms of high magnetic moment, superparamagnetic behavior at room temperature and photocatalytic response under visible light.

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# Tunable first order transition in La(Fe,Cr,Si)<sub>13</sub> magnetocaloric compounds

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Recently proposed criterion based on the field dependence of the isothermal entropy change ( $\Delta s_T(T, \Delta H) = a(T)\Delta H^{n(T,H)}$ ) can be used for identifying first order phase transition (FOPT) [1], whereby the field exponent  $n$  yields an overshoot of  $>2$ . In addition,  $n$  at transition temperature ( $n(T_{trans})$ ) can further reveal the critical composition of the crossover of FOPT to second order phase transition (SOPT) as  $n(T_{trans}) < 0.4$  indicates FOPT,  $n(T_{trans}) > 0.4$  for SOPT and  $n(T_{trans}) = 0.4$  as the crossover [2]. In this work, we report that Cr-doped La(Fe,Si)<sub>13</sub> compounds (for  $x \leq 0.6$  in LaFe<sub>11.6-x</sub>Cr<sub>x</sub>Si<sub>1.4</sub>) are found to exhibit a maintained magnetocaloric response despite the dopant diluting the overall magnetic moment [3]. The magnetocaloric response is retained up to  $x \approx 0.3$  due to the increment of the FOPT-character (which is characterized by the reduction of  $n(T_{trans})$  values). For higher Cr content, the series exhibits a crossover of FOPT to SOPT. These features are presented in Figure 1. Moreover, a direct relation between the first order character and the hysteresis is observed, in agreement with the performed analysis. These results illustrate the importance of the nature of phase transition in both magnetocaloric response as well as its compositional dependence.

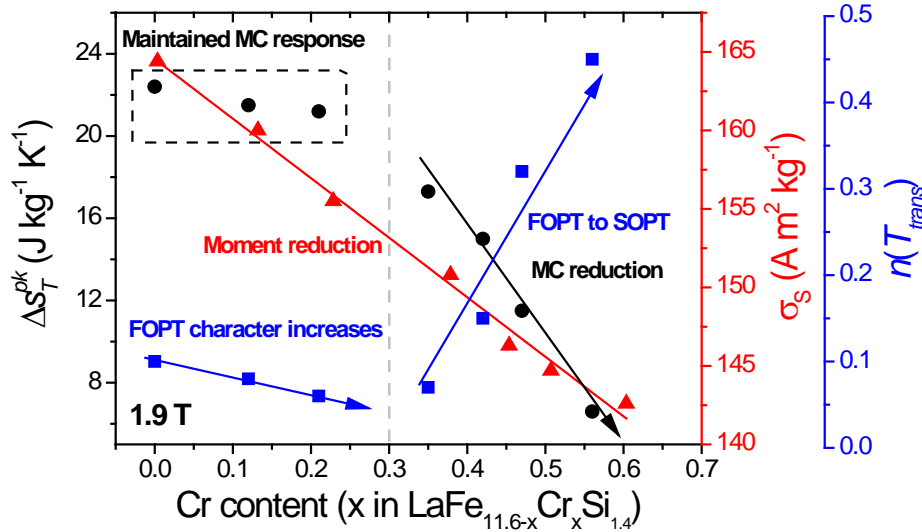


Figure 1. Compositional dependence of maximum isothermal entropy change ( $\Delta s_T^{pk}$ ), saturation magnetization ( $\sigma_S$ ) and exponent  $n$  at the transition temperatures in La(Fe,Cr,Si)<sub>13</sub>.

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*Index Terms* — Magnetocaloric materials, Thermomagnetic phase transitions, La(Fe,Si)<sub>13</sub> compounds.

# The role of shape anisotropy in the magnetosome chain

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Magnetotactic bacteria (MTB) are a diverse group of microorganisms with the ability to orient and migrate along the geomagnetic field due to the presence of a chain of magnetic nanoparticles called magnetosomes. In particular, *Magnetospirillum gryphiswaldense* MSR-1 species synthesizes cubo-octahedral shape magnetite ( $\text{Fe}_3\text{O}_4$ ) nanoparticles with a mean diameter of 45 nm. These magnetosomes are arranged forming a slightly bent helical-like shape chain [1]. This arrangement results from the interplay between an elastic recovery force and the dipolar magnetic interaction between magnetosomes, ruled by the orientation of the magnetosomes magnetic moment. In that work, I. Orue et al [1] suggest that the magnetic moment of each magnetosome is tilted  $20^\circ$  out the [111] crystallographic easy axis of magnetite.

The main aim of the present work is to understand the origin of this uniaxial anisotropy that should come from the competition between the crystalline anisotropy of the magnetite and a shape anisotropy of the magnetosome. We have proceeded in the following way. First, we have studied the shape of the magnetosomes using electron cryotomography (ECT). The tomographs show that the magnetosomes are not ideal truncated octahedrons (Figure 1a), a 10% elongation of the [1-11] axis and a 7.5% of the [001] axis were found. Second, we have simulated the magnetostatic energy of one single particle with this deformation, using finite element methods. From the simulations, we have seen that the distortion found by ECT is enough to deviate the magnetic moment  $20^\circ$  with a density energy landscape typical of a uniaxial anisotropy system with an anisotropy constant  $K=7 \text{ kJ/m}^3$  (Figure 1b). Finally, we were able to simulate the AC hysteresis loop of the magnetosomes considering both crystalline and uniaxial shape anisotropies and dipolar interaction between the magnetosomes (Figure 1c).

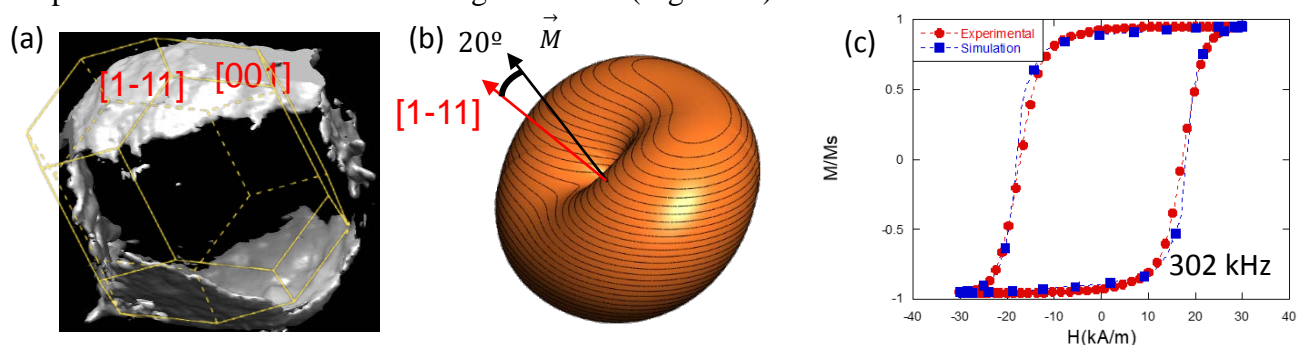


Figure 1. (a) ECT 3D reconstruction of one magnetosome. (b) Shape anisotropy energy landscape of the magnetosome. (c) AC hysteresis loops measured at 302 kHz of bacteria in water. Experimental data (red circles) and simulated data (blue squares).

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**Keywords** — Magnetotactic bacteria, magnetic anisotropy, tomography.

# Variation on the magnetic properties by high energy ball-milling in Ni<sub>45</sub>Co<sub>5</sub>Mn<sub>35</sub>Sn<sub>15</sub> metamagnetic shape memory alloys

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The metamagnetic shape memory alloys, based on Ni<sub>2</sub>-Mn-Z (Z=Sn, In, Sb), are one of the smart material studied in the recent years due to their potential application in magnetic refrigeration and sensors. Both applications are focalized in the effect that those alloys presents around martensitic transformation as magnetocaloric effect in magnetic refrigeration and giant magnetoresistance in sensors. The martensitic transformation happens between ferromagnetic high symmetry austenitic phase and low symmetry martensitic phase with weak magnetic structure, for this reason in the martensitic transformation there is a variation on the magnetization that is the most important part to introduce these alloys in their applications. The microstructural parameters (long atomic order, internal stress, grain size, vacancies and defects) play a crucial role in the magnetostructural properties.

Here, we have studied how the ball-milling time affects the magnetostructural properties and to martensitic transformation in Ni<sub>45</sub>Co<sub>5</sub>Mn<sub>35</sub>Sn<sub>15</sub> alloy. With X-rays powder diffraction, we have calculated the microstructural parameters induced mechanically as grain size and internal stress and with neutron powder diffraction we have checked if the ball-milling treatment produce atomic disorder.

# Production and microstructural and magnetic characterization of the composition $\text{MnCo}_{0.8}\text{Fe}_{0.2}\text{Ge}$ obtained by mechanical alloying

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Current technological applications require a high degree of control in the composition and the structure of materials. Both characteristics are intertwined particularly when the aim is to obtain intermetallic phases. The use of thermal treatments at high temperature and long time is necessary when the functional properties of interest are restricted to certain phases present in a narrow composition range and stable in a certain temperature range [1]. However, mechanical alloying allows obtaining nanometric size precursor systems whose homogeneity can reduce both time and treatment temperature. Half-Heusler alloys have been proposed as interesting systems for magnetocaloric studies [2]. The absence of rare-earth metals and the tunability of the temperature and order character of the transition are very attractive in these compositions. In this work, it has been possible to reduce thermal treatment from several days to a few minutes and from 850 °C to 450 °C for the precursor  $\text{MnCo}_{0.8}\text{Fe}_{0.2}\text{Ge}$  obtained by mechanical alloying. Microstructure is studied by X-ray diffraction. Mössbauer spectroscopy ( $^{57}\text{Fe}$ ) is used to follow the changes in the Fe environments [3]. Thermomagnetic measurements allow us to analyze the magnetocaloric effect and to compare the results with those obtained from conventionally annealing treatments.

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*Keywords:* intermetallic, mechanical alloying, microstructure, magnetic properties.

## Acknowledgements

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# The Mechanical and Magnetic Properties of Mn-Al-C-Cu Alloys

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The  $L1_0$  structured  $\tau$ -phase of the Mn-Al-C system gained interest for the potential use as a rare earth (RE) free hard magnetic material. Warm extrusion is a promising route to optimise the extrinsic magnetic properties of MnAl-C alloys; however, the high stresses involved reduce the lifetime of the extrusion tools, which in turn raises the production costs [1]. It has been reported that Cu addition can reduce the force needed for extrusion without negatively affecting the magnetic properties [1] but the mechanism behind this effect was not shown. In the current work, the effect of Cu additions on the microstructure, mechanical and magnetic properties of MnAl-C alloys in various states has been investigated in detail.

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# Remanence Improvement in Exchange-Decoupled Composites owing to Dipolar Interactions

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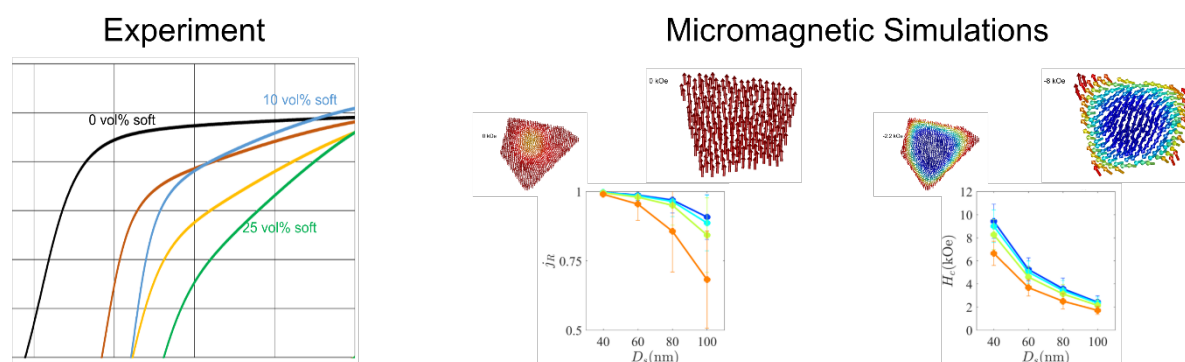
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The theory on exchange-spring magnets, published by Kneller and Hawig in 1991, heralded the next big leap forward in the field of advanced permanent magnets since the discovery of Nd<sub>2</sub>Fe<sub>14</sub>B in 1982. The theory predicted that a magnet prepared by combination of a hard and a soft magnetic phase would exhibit an enhanced remanent magnetization,  $M_r$ , as long as the two phases were mutually exchange-coupled [1]. However, accomplishing an actual improvement using the exchange-coupling strategy has proven rather challenging when it comes to real systems.

When large particles of soft phase are strongly exchange-coupled to a hard phase, a magnetic softening is induced, instead of the desired hardening [2]. However, moderate  $M_r$  improvements have been detected in exchange-decoupled composites with soft phases of large dimensions [3]. In exchange-decoupled systems, the dominant magnetic interactions are magnetodipolar [4].

Therefore, exploiting the dipolar interactions between exchange-decoupled hard and soft phases arises as an alternative strategy to prepare magnetic composites with an enhanced performance [5]. The efficacy of the method relies on controlling the magnitude of the self-demagnetizing fields, which in turn depend strongly on the size and shape of the soft phase. Here, exchange-decoupled SrFe<sub>12</sub>O<sub>19</sub> (hard)/ $\alpha$ -Fe (soft) composites have been prepared. The influence of the size of the soft phase has been experimentally studied, covering a range between 25 nm and 11  $\mu$ m. Micromagnetic simulations have been carried out for the specific system in order to understand the spin dynamics inside the soft grains (see Figure). Regardless of the soft phase dimensions, the composites show a larger  $M_r$  than predicted by the theory.



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*Index Terms* — SrFe<sub>12</sub>O<sub>19</sub> (hard)/ $\alpha$ -Fe particles (soft), Remanent magnetization, Exchange-coupled, Micromagnetic simulations

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# Analytic formulae for moving permanent magnets

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The study of devices featuring permanent magnets can be performed by numerical (e.g. finite element analysis) or analytical methods. However, numerical simulations of moving magnets can be time consuming and complicate the optimization. On the other hand, simplifications and limitations of analytical formulae have to be clearly defined in each case. As part of a larger work, a set of analytical expressions is derived to calculate the varying magnetic field detected by a sensor attached to an electromagnetic energy harvester based on magnetic levitation. Then relationships to other physical magnitudes of the whole system are determined. This simplified theoretical analysis is enough to explain why and how a magnetic sensor can be used to measure the vibration amplitude of a surface. It also matches experimental data, qualitatively and to some extent quantitatively.

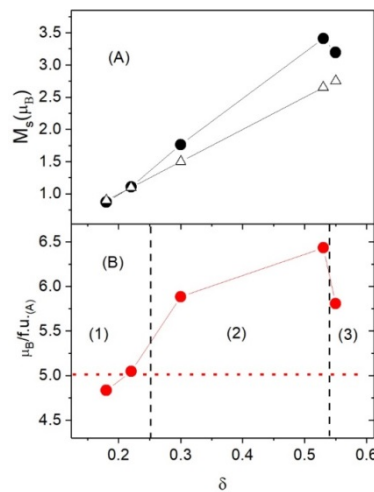
*Index Terms* — Permanent magnet, magnetic field, analytical calculation.

# Structural and magnetic properties of zinc ferrite nanostructures

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Massive zinc ferrite has a normal spinel structure (with all  $Zn^{2+}$  cations in the tetrahedral (A) sites, while  $Fe^{3+}$  cations occupy the octahedral (B) ones), and it behaves paramagnetic at room temperature. It has been reported that synthetic and processing methods can have a significant influence on the physical properties of spinel since it can produce a metastable disordered structure. In this case, the structural formula must be written as  $(Zn_{1-\delta}Fe_{\delta})[Zn_{\delta}Fe_{2-\delta}]O_4$ , where  $\delta$  represents the degree of inversion (defined as the fraction of A sites occupied by Fe cations). This redistribution of  $Zn^{2+}$  and  $Fe^{3+}$  cations leads to the onset  $Fe^{3+} - Fe^{3+}$  magnetic interaction between A-B sublattices. Hence the importance of establishing the relationship between structural parameters and magnetic properties. In this work three main routes of synthesis to obtain zinc ferrite have been carried out: Ceramic, Mechano-synthesis and Sol-Gel methods. In each of them, the synthesized ferrites have been characterized to know the structural and magnetic properties. Thereafter, cationic order or disorder is induced by mean of thermal annealing or ball milling. So, although the synthesis method determines the starting structural properties, these properties can be varied by thermomechanical process (grinding and thermal annealing), and the required magnetic properties can be tuned to get the best results for different applications.



**Figure:** (A) saturation magnetization per Bohr magneton as a function inversion degree  $\delta$  (black circles) and saturation calculated as  $M_s = \delta(5\mu_B)$  (open triangles). (B) Saturation magnetization per

ferromagnetic unit (f.u.) at A sites. The regions (1), (2) and (3) indicate FiM+AFM+SG, FiM and FiM+AFM phases, respectively.

## Magnetic field influence on magnetomechanical coupling of partially bistable magnetic materials

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Amorphous magnetic microwires (AMW) are used for several applications, mainly for the development of sensors. AWM are continuous filaments with a magnetic core covered by a glassy outer shell. Generally, the total diameter is less than 100 μm and the diameter of the metallic core is between 4 to 60 μm. This material is obtained by Taylor's technique and its magnetostriction constant can be positive or negative depending on the Fe/Co percentage.

FeSiB based magnetic microwires and ribbons with positive magnetostriction constant present high magnetomechanical coupling that allows efficient conversion between magnetic and elastic energies and vice versa.

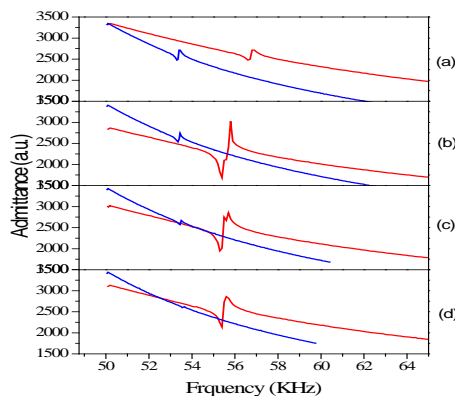
The magnetic material used in this work are ribbons and glass coated microwires. We obtain a resonant frequency corresponding to an optimal applied magnetic field. A large study will be present for different concentration of sensor's environment.

The aim of this work is to study the evolution of resonance frequency of our magnetoelastic element immersed in different solutions.

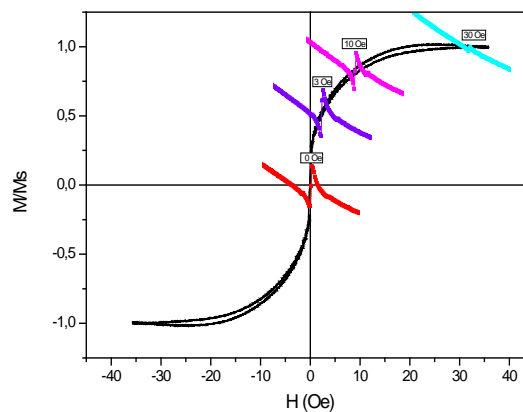
It should be remarked that the amplitude of the resonance frequency depends on the viscosity but the frequency change is related to the additional mass added to the sensor surface.

The fundamental resonant frequency under longitudinal vibration is expressed as:

$$f = \frac{1}{2L} \sqrt{\frac{E}{\rho(1-\nu)}}$$



**Fig.1:** Spectrum resonance for (–) wire, (–) ribbon of 1mm of width and for four different value of applied DC field.  
(a) H=0 Oe, (b) H=1 Oe, (c) H=3 Oe, (d) H=9 Oe



**Fig 2:** Hysteresis loops of a 3.5 cm length of glass coated microwire (dm=60um, Dgl=100um) and resonance frequency evolution

# MFM-KPFM characterization of magnetic nanocomposites for bioapplications

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Magnetic nanostructures have been used in different applications in biomedicine and life science such as imaging diagnosis, drug delivery, hyperthermia and molecular detection [1]. Moreover, the effect of different physical stimuli in the cell culture has been studied. In particular, conductive or piezoelectric polymers have been used for tissue engineering applications, due to their ability to create a beneficial electroactive microenvironment to the cells [2, 3]. In this work, a fundamental study of the magnetic and electrical properties of a magnetoelectric nanocomposite is presented, which has shown applications in biomedicine [4].

The composite material is prepared by mixing magnetic nanowires 60 nm in diameter partially embedded into an AAO membrane with interpore distance of 105 nm and a piezoelectric polymer poly(vinylidene fluoride) (PVDF) thin film. Magnetic Force Microscopy-Kelvin Probe Force Microscopy (MFM-KPFM) combined system [5] has been used to characterize the electrical and magnetic properties of this material at the nanoscale. Thanks to this combined system, it is possible to distinguish the magnetic signal coming from the magnetic nanowires and the surface potential of the PVDF layer that varies with its thickness as shown in Figures 1 and 2.

*Index Terms* — Magnetic Force Microscopy, Kelvin Probe Force Microscopy, magnetoelectric nanocomposites.

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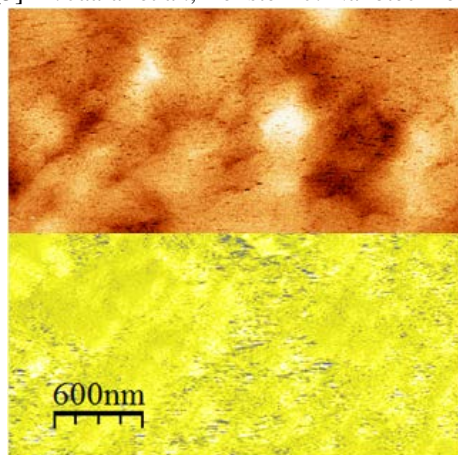


Figure 1. MFM (upper image) and KPFM (lower image) corresponding to a region with a thin polymer layer covering the partially embedded nanowires.

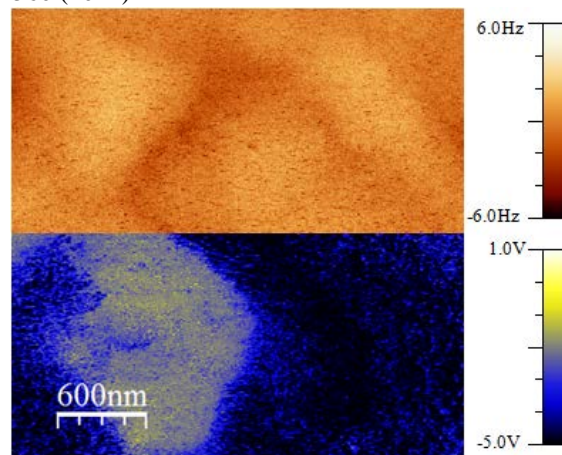


Figure 2. MFM (upper image) and KPFM (lower image) corresponding to a region with a thicker polymer layer covering the partially embedded nanowires.