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Magnetic and structural characterization of Fe-Ni films for high precision field sensing

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In this paper, results are presented concerning structural and magnetic characterization of magnetic field sensing core Fe-Ni films utilizing super-paramagnetic-like rotation of magnetization on the film plane in order to minimize the magnetic noise and improve the system sensitivity. The rotating vector of magnetization was realized by transmitting sinusoidal and cosine fields on the film plane, having amplitude much larger than the anisotropy field of the film. Kerr microscopy studies and magnetoresistive measurements have been realized allowing the evaluation of the developed sensing cores. Furthermore XPS analysis helped in correlating the surface structure with the properties of the developed materials. In this way, the selected sensing cores have been tested with respect to the applied field, illustrating acceptable levels of sensitivity and noise. Such an arrangement can be considered as promising for the development of new types of high precision monolithic CMOS compatible field sensors.

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1. Introduction

In today's sensing technology the anisotropic magnetoresistant effect is widely and commercially used, especially for magnetic field and linear or angular sensing [1]. Another technique employs the giant magneto-impedance effect [2-4], which has already resulted in commercial products. Giant magneto-resistance has also been used in the past as field sensor core [5, 6]. Other effects like the Hall effect [7] has also been employed commercially with a rather limited sensitivity. Experimental trials employing the magnetostriction effect [8] have also been tried.

Amongst the factors limiting sensitivity of magnetometers, the ones related to the thermodynamic non-equilibrium state in the remagnetization cycle of the sensing element are the most significant. Formation of non-saturated regions of the sensitive element, pinning effects in domains and domain walls, inhomogeneities at dislocations and other defects contribute to the instability and irreversibility process of the magnetization procedure, causing magnetic noise in a sensing element based on magnetic materials. Transition processes during the establishment of the equilibrium can also produce irregularities. All aforementioned factors cause a sensitivity decrease and the 1/f type of noise that is typical for fluxgate magnetic field sensors. In these systems an adequate evaluation of the sensitivity threshold of a sensor on the basis of the material parameters is rather problematic. There is a trade off between device sensitivity and measurement repeatability; the anisotropy field H_k affects both parameters in an inverse manner [9].

Many of these problems can be avoided by utilising magnetization rotation in saturated single-domain magnetic films, in a way to suppress Barkhausen noise [10]. Using the ferro-dielectric yttrium iron garnet material enables one to avoid eddy current losses, which limit the excitation frequency - and thus the magnetometer sensitivity - and which are also an additional source of noise. Conditions of simultaneous measuring of three orthogonal magnetic field components by the yttrium iron garnet single-crystal film have been established due to the cubic anisotropy of a single-crystal yttrium iron garnet film which allows for designing 3D magnetometers [11].

The employment of AMR film resistors as Fluxgate devices has also been proposed [12]. The film magnetization M rotates under the influence of a harmonic excitation field. Angle φ is modulated by the axial components h_x and h_y of the ambient magnetic field and, thus, film resistance becomes a phase modulated waveform. The devices seem to have excellent linearity, repeatability and directionality along both x and y axes. Their resolution is limited only by Barkhausen noise due to magnetization pinning, which in the case of single-domain films is very weak. The design of sensors with resolution in the range of $\Delta H \sim 10^{-4} H_k$ is possible with standard Permalloy films ($H_K \sim 400$A/m). The employment of magnetic annealing and/or isotropic alloys (Supermalloy) would improve the performance significantly.

The motivation of the current research activity is the design, development and optimization of a magnetometer able to perform magnetic noise in the region of about 1 pT at 1 Hz at room temperature; such a principle idea has already been presented [12], while the present paper contributes towards the improvement of the characteristics of such a magnetometer, due to surface characterization.

The main idea behind such a sensor is that Barkhausen noise does not exist, since the sensing film is under rotating magnetization with fields much larger than the anisotropy field of the material.

2. Sensor core development and characterization

The development of the sensing cores of the conceived magnetometer was based on the Ni-Fe family of alloys due to the well known soft magnetic properties. A variety of thin films of the Ni_xFe_{1-x} composition was developed, in order to determine the optimum alloy composition and thickness as well as the dependence of film performance on the history of the films. The optimum composition, thickness and annealing history of the films should minimize the noise of the field sensor. Therefore, the critical points of interest were the as low as possible coercive and anisotropy fields H_C and H_k respectively, along the plane of the sample. The samples were made by mother alloy evaporation with electron beam under vacuum of the order 6×10^{-6} mbar. The mother alloy compositions were Ni_xFe_{1-x} with x = 0.4, 0.6, 0.75, 0.80, 0.85 as well as the supermalloy $Ni_{77.5}Fe_{19}Mo_{3.5}$ phase. Various thicknesses of the films have been realized, from 30 nm to 100 nm. The technology to minimize the instability of composition homogeneity has been achieved in the used e-beam evaporator, by controlling the evaporation rate. Such a problem appeared due to the fact that the concentration of the deposited samples was not stable in depth, because of the small difference in evaporating temperatures of the two elements (2732^{0}C and 2750^{0}C respectively).

XRD studies could not be performed for a systematic and detailed structural study of the developed films, since the penetration depth is much greater of the sample's thickness. Therefore, X-ray photoelectron spectroscopy (XPS) was used to study the surface and near surface composition and chemical state of the films. XPS studies were performed by using both Al Kα (hν=1486.6 eV) and Mg Kα (hν=1253.6 eV) radiation, in order to acquire chemical state information from different depths in the sample. Figs. 1, 2 and 3 show XPS results of the samples analysed. The results showed that the outermost surface is dominated by Fe oxide of a thickness of ~ 40Å. High resolution Ni 2p spectra showed a depletion of Ni on the outermost surface and an enhancement of its presence after a depth of ~ 25Å. Nickel was found almost exclusively in metallic form, as expected from the thermodynamic stability of the Fe and Ni oxides. The interesting outcome of such a study, concerning films developed and studied up to this moment, was that the surface oxidation had a rather uniform depth of 35Å, which was more or less repeatable in all samples. This experimental evidence suggested a uniform flat ferromagnetic film below the protecting surface oxide, promising soft magnetic response of the samples.

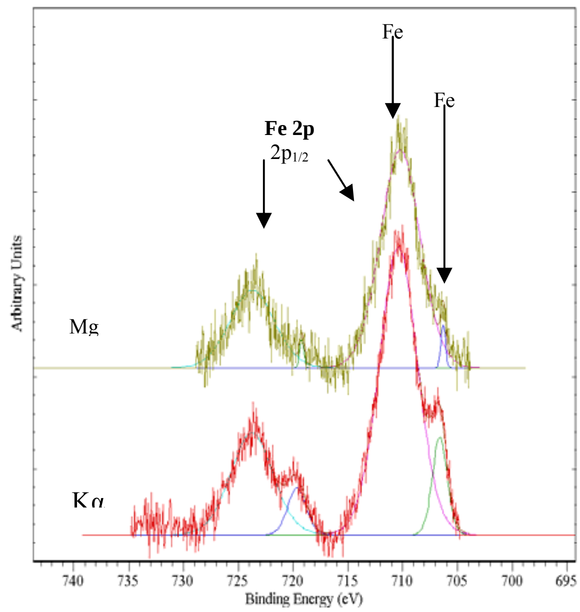

Fig. 1. High resolution Fe 2p spectrum showing that the outermost surface is dominated by Fe oxide. Shirley type of background subtracted.

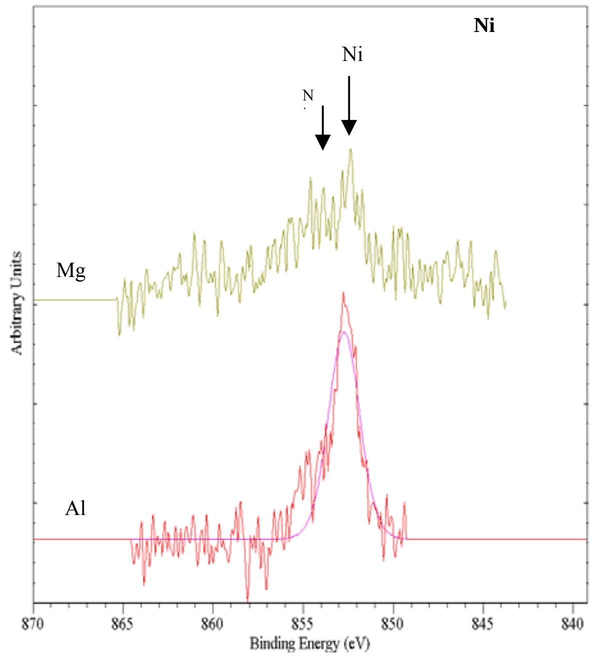

Fig. 2. High resolution Ni 2p spectra showing depletion of Ni on the outermost surface Shirley type of background subtracted.

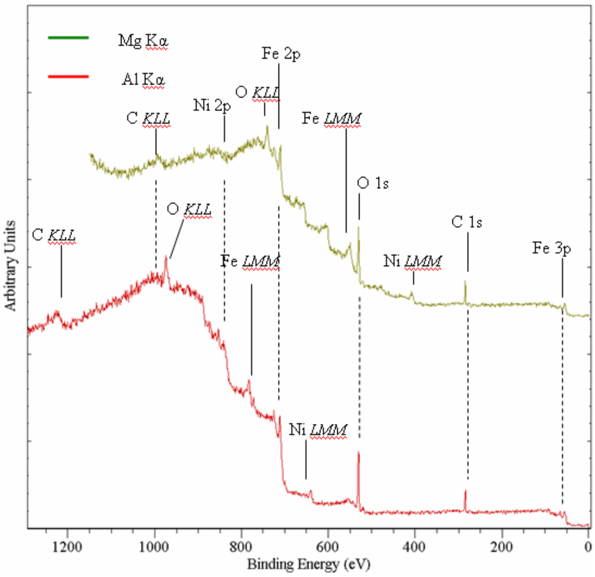

Fig. 3. XPS survey scans showing a highly increasing background on the high binding energy side indicating presence of oxide and surface contamination.

Having experimentally achieved a surface oxide protecting layer and a relatively smooth ferromagnetic layer, magnetic, magneto-optic and magneto-resistive characterization has been performed. Static and ac magnetometry illustrated that the softest magnetic behaviour was obtained for the $Ni_{75}Fe_{25}$ composition of films, for the developed and measured films. This fact has also been proven by using Kerr effect magnetic microscopy. In Fig. 4, the best obtained performance using Kerr effect measurements is illustrated concerning $Ni_{75}Fe_{25}$, measured in the as – manufactured state and illustrating ~100 A/m and 300 A/m coercive and anisotropy fields respectively. Similar effect has also been observed by performing magneto-resistance measurements. As illustrated in Fig. 5 and 6, demonstrating magneto-resistive measurements of as – manufactured films on the X and Y axis of the film, a non –stable MR performance has been observed, suggesting an isotropic behaviour of the tested film. The magnetoresistance measurements have been taken by the 4-point technique with 1 μOhm resolution. As seen in Figs. 5 and 6, the permalloy thin film reaches saturation at ~ 300 A/m, which is in agreement with the magnetic and magneto-optic studies. The abnormality observed in ~ 200 A/m, before the element reaches saturation is due to magnetization remanence, which vanishes as field gets larger. Although magnetic softness and isotropic behavior was observed for compositions close to the small magnetostriction compositions, the optimum thickness of the films has been determined by the most sensitive MR measurements. According to these measurements, a thickness of 30 nm, apparently maximized the MR response, in the films prepared up to this moment, maintaining their soft and isotropic magnetic behavior.

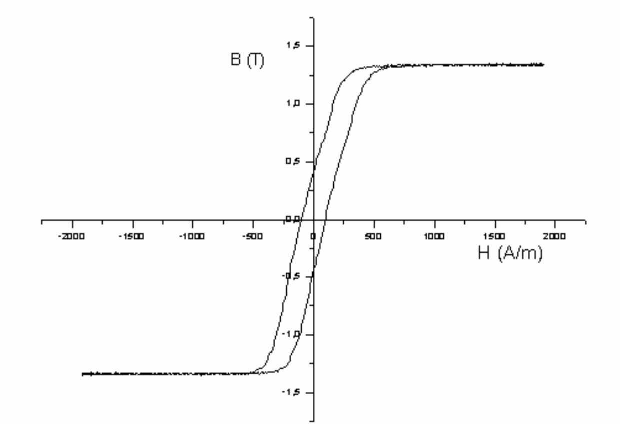

Fig. 4. Permalloy hysteresis loop obtained by Kerr microscopy.

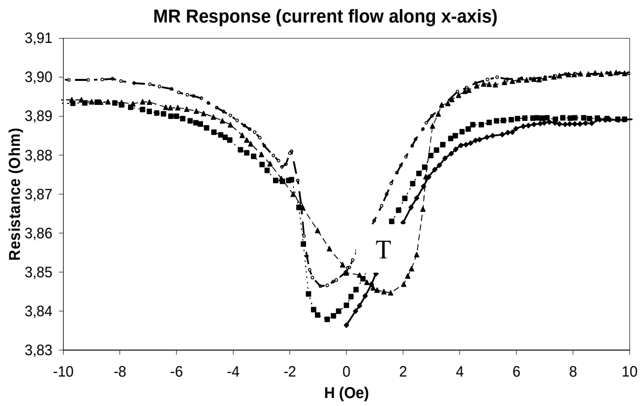

Fig. 5. MR response (Current flow along x-axis).

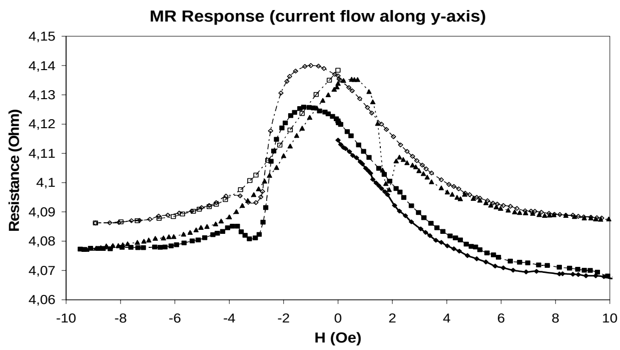

Fig. 6. MR response (Current flow along y-axis).

Furthermore, magnetic annealing improved the magnetic softness, but also increased the on plane anisotropy of the films. Magnetic annealing also resulted in better but anisotropic MR performance. This cannot be acceptable for the under development sensors, since the isotorpic magnetic behaviour on plane allows minimization of Barkhausen noise during on plane rotation of magnetization.

The above mentioned experimental results and correlation between structural studies and magnetic properties of the under test films, concerning the developed films up to this moment, resulted in the conclusion that magnetic thin films of the $Ni_{75}Fe_{25}$ composition having 30 nm thickness and used in the as – manufactured state, may be promoted as the most isotorpic and stable sensing core films. This is due to the fact that the in plane anisotropy is maintained low, which may allow the magnetization of the core to follow the rotating excitation field closely. Taking all these facts into account, it may be predicted that a rotating field of ~500 A/m may be appropriate to keep the 30 nm thick as-cast $Ni_{75}Fe_{25}$ film in constant circular saturation without Barkhausen jumps, thus minimizing the magnetic noise of the film.

3. Sensor core dependence on field

The used experimental set-up for the galvanic response of the films under rotating field was as described in [12]. The rotating excitation field was applied on the film plane by using two orthogonal coils offering identical magnetic fields. The coils were wound around the core and excited by two phase-shifted sinusoidal fields namely $H_x=H_o\cdot sin(\omega_o\cdot t)$ and $H_y=H_o\cdot cos(\omega_o\cdot t)$. High sensitivity MR heads with precision standard multimeters were used to measure the excitation fields. A signal integrator was transforming the sinus signal to cosine, so the superposition of the field of the two orthogonal coils was generating the rotating magnetic field vector used for excitation. For practical testing reasons, the rotating frequency was set at 1 KHz. Excitation field amplitude H_o was fixed set to 800 A/m instead of 500 A/m, to ensure that magnetization was force to complete saturation. The whole set-up was placed in a Faraday chamber for magnetic shielding, to minimize the ambient field effect, such as earth's field. In this way the magnetization vector in the film plane rotates smoothly, thus generating minor and repeatable magnetic noise and therefore error.

The used film was the previously described 30 nm thick as-cast $Ni_{75}Fe_{25}$ film with no barber pole configuration as suggested in the past [13]. So, even values of the sinus and the cosine components are suitable to circularly excite the thin film, as seen in Fig. 7. From this figure it can be seen that magneto-resistance is a harmonic time-function (signal) that alternates with frequency $\omega = 2\omega_0$ maximizing the resistance alternation every time the radius equals to π. Additional ambient DC magnetic field evokes phase delay to the resistance signal that can be measured at the time instances when magnetization crosses the *x*-axis.

The signals related to *x*- and *y*- *c*omponents of ambient field are orthogonal having a constant *90°* phase difference. Magnetization and magnetic field vectors are illustrated in Fig. 8, suggesting significant phase modulation.

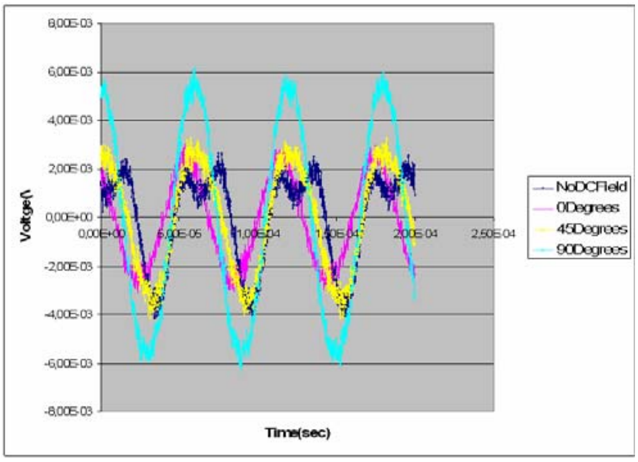

Fig. 7. Sensor output under rotating excitation field with equal components on x- and y- axis. The magnetization rotates uniformly and alternations occur when external DC field is applied.

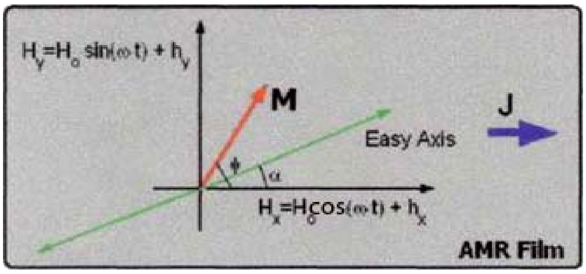

Fig. 8. Definition of magnetization-, current-, and in-plane magnetization field-vectors.

As reported in the past [14], the x- and y- components of the external magnetic field applied on the sensor, affect even and odd zero crossings of the sensor output-voltage (Fig. 7). Thus, by locking-in different zero crossings of the excitation sine wave, the two components of the external field can be measured. Actually, the instances of interest are zero crossings and maxima of the excitation sinus waveform. It has also been proven experimentally that the use of a triangle waveform leads to better sensitivity of the output, due to the intrinsic design of the lock-in amplifier.

Thus, using lock-in amplifier to measure phase difference between excitation field and output voltage corresponding to resistance change, precise magnetic field sensing can be realized. The actual schematic of the electronic circuitry, allowing such measurements due to filtering techniques is illustrated in Fig. 9.

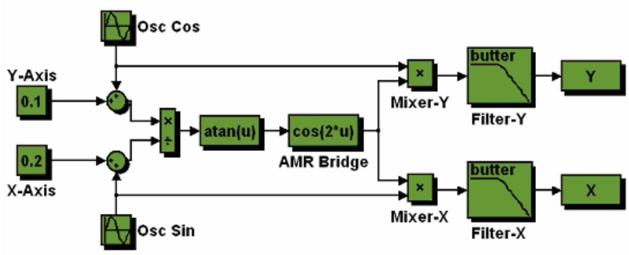

Fig. 9. Schematic of the setup that has been developed for the measurement of 2D weak DC magnetic fields.

A typical phase difference dependence on field is illustrated in Fig. 10. As expected the thin film sensing element exhibits negligible repeatability error, despite the fact that the MR response of the employed Permalloy thin-film was quite unpredictable as showed in the previous chapter. The forced magnetization rotation suppressed in fact both magnetic noise and repeatability error. The final field sensor accuracy is also dependent on the system electronics and requires further exploitation.

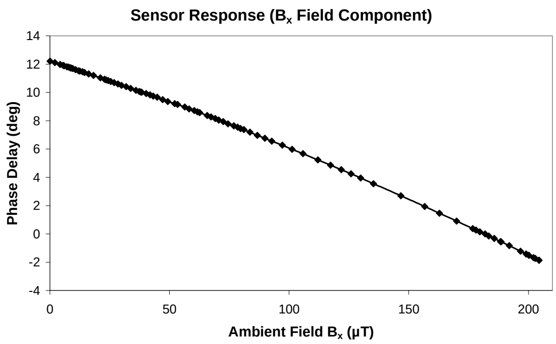

Fig. 10. A typical sensor response (B_x field component demonstrated).

The above mentioned sensitivity is absolutely dependent on the rotation – excitation field intensity. Setting the excitation field H_o field 500 - 800 A/m the sensitivity was exhibiting the above mentioned values. Lowering such excitation field below 450 A/m the voltage output suffered from significant instabilities attributed to Barkhausen noise, thus lowering the sensitivity to ~ 1 nT. Furthermore, using higher amplitude of excitation field, very low dc fields were not measurable, because they cannot influence significantly the rotation of magnetization of the material and thus the output waveform. Thus, as it was proven experimentally, excitation amplitudes of 500 – 800 A/m were the optimum excitation field for the current setup and material of the sensing core.

4. Conclusion and future work

A new 2-D hybrid AMR/Fluxgate sensor for DC magnetic field measurements has been presented that exhibits high sensitivity, low magnetic noise, and negligible repeatability error. Moreover, compared with standard Fluxgates, it does not require a receiving coil to function. The sensor design is compact and robust and its miniaturization is straightforward by means of standard microelectronic technologies. Future work is underway for the realization of films of different composition, thickness and annealing history in order to evaluate and model their performance as sensing cores of the under development field sensor. Accordingly characterization in very low fields is also under way for the determination of the sensitivity limits in the frequency domains.

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