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Nanowires of Fe/MgO/Fe Encapsulated in Carbon Nanotubes

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Abstract

Nanowires of tunneling magnetoresistance (TMR) were synthesized using magnetron DC/RF sputtering by filling Fe/MgO/Fe inside vertically grown and substrate-supported carbon nanotubes. Nanocolumns of Fe/MgO/Fe TMR were synthesized using glancing angle deposition. The magnetic properties of nanowires, nanocolumns and planar nanometric thin films of Fe/MgO/Fe showed similarities including twofold magnetic symmetry. Nanowires of Fe/MgO/Fe showed improved magnetic properties, in particular its coercive field, which is 754% higher than planar thin films of Fe/MgO/Fe. A macroscopic phenomenon that can be explained only by quantum mechanics is TMR, where electrical current can flow across a nanometric thin insulator layer between two electrodes when an external magnetic field is applied parallel to the trilayer system. Coherence in the TMR effect is paramount to make spintronic devices. Nanowires possess shape anisotropy, which can play an important role in coherence.

Keywords: nanowires, Fe/MgO/Fe, magnetoresistance, anisotropy, sputtering

1. Introduction

In TMR, electrical current flows across a barrier of nanometric thin insulator layer between two ferromagnetic metal electrodes when an external magnetic field is applied parallel to the trilayer surface. TMR is one of the few examples of macroscopic quantum mechanical phenomena that have no classical explanation. TMR has a wide array of applications including magnetic random access memory for futuristic quantum computer and ultrasensitive sensors [1]. TMR is the basic building block of magnetic tunnel junctions (MTJs); specifically Fe/MgO/Fe TMR having one of the highest magnetoresistance (MR) ratio is of current interest as evidenced by

several studies of its magnetic properties [2–7]. Theory predicts several thousand percent MR ratio for Fe/MgO/Fe TMR trilayers when an interface is modeled to possess an abrupt change from Fe to MgO without any diffusion at the interface [3]. When diffusion at the interface is introduced in the theoretical modeling by way of interface oxidation, the MR ratio dropped to 1000% [2]. First-principle modeling showed that diffusion at the interface plays a major role in decreasing the calculated value of the MR ratio to the extent when only 16% of Fe is replaced by Mg and vice versa; the MR ratio agreed with experimental values for higher values of the insulator (MgO) thickness [4]. The noncollinear nature of the atomic moments with the bulk magnetization at the interface of Fe and MgO will perhaps affect the net anisotropy, or it may be a spin scattering locality [5]. Experimental measurements showed that single-crystal Fe/MgO/Fe has an MR ratio of 180% at room temperature and 247% at 20 K [6]. It was also shown experimentally that for crystalline MgO (001), the barrier between the electrodes resulted in a 220% MR ratio at room temperature [7]. The symmetry of electronic wave functions plays a paramount role in coherent spin-polarized tunneling, which gives rise to enormous TMR effects. Shape anisotropy plays an important role in coherent spin-polarized tunneling of electrons across the insulator barrier; thus, switching the geometry of TMR from planar films to nanowires will increase the coherence thus increasing the MR ratio [8, 9]. The importance of shape anisotropy in ferromagnet/insulator/ferromagnet trilayer structures in particular for Fe/MgO/Fe and the effect of encapsulation of a TMR by carbon tubes are discussed in this chapter based mainly on magnetic measurement results and subsequent interpretations.

TMR-based magnetic tunnel junctions are theoretically expected to exhibit an extremely high MR ratio due to coherent tunneling [2]. When the coherency of electron wave functions is conserved during tunneling, only conduction electrons whose wave functions are totally

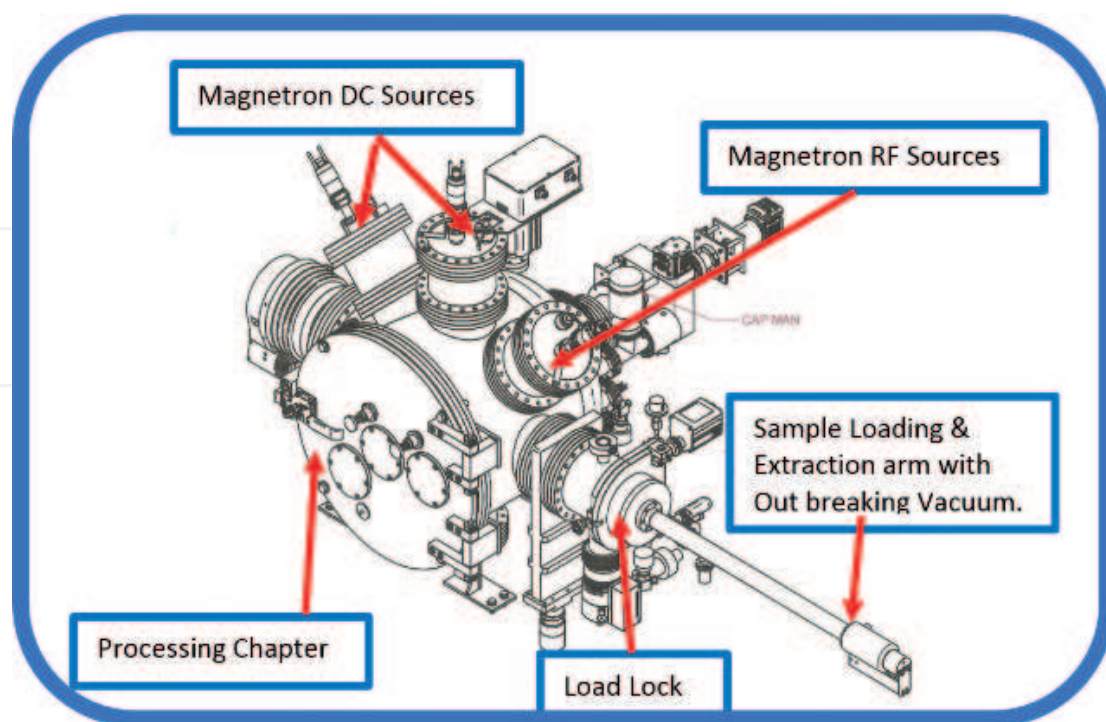


Figure 1. Magnetron sputtering tool with 3 DC/1 RF sources and a load-lock.

symmetrical with respect to the barrier-normal axis are connected to the electronic states in the barrier region and have significant tunneling probability. The MR ratio is defined as follows:

$$\text{MR ratio} = (R_{\text{ap}} - R_{\text{p}}) / R_{\text{p}}$$

where R_{p} and R_{ap} are the tunnel resistance when the magnetizations of the two electrodes are aligned in parallel and antiparallel, respectively (**Figure 1**).

A TMR system reveals unique properties with attractive effects for technological applications. Besides giant and tunneling magnetoresistance, it presents other remarkable effects such as antiferromagnetic exchange coupling, oscillatory behavior of exchange coupling and biquadratic exchange couplings. Nanometric ultrathin films exhibit an out-of-plane uniaxial surface

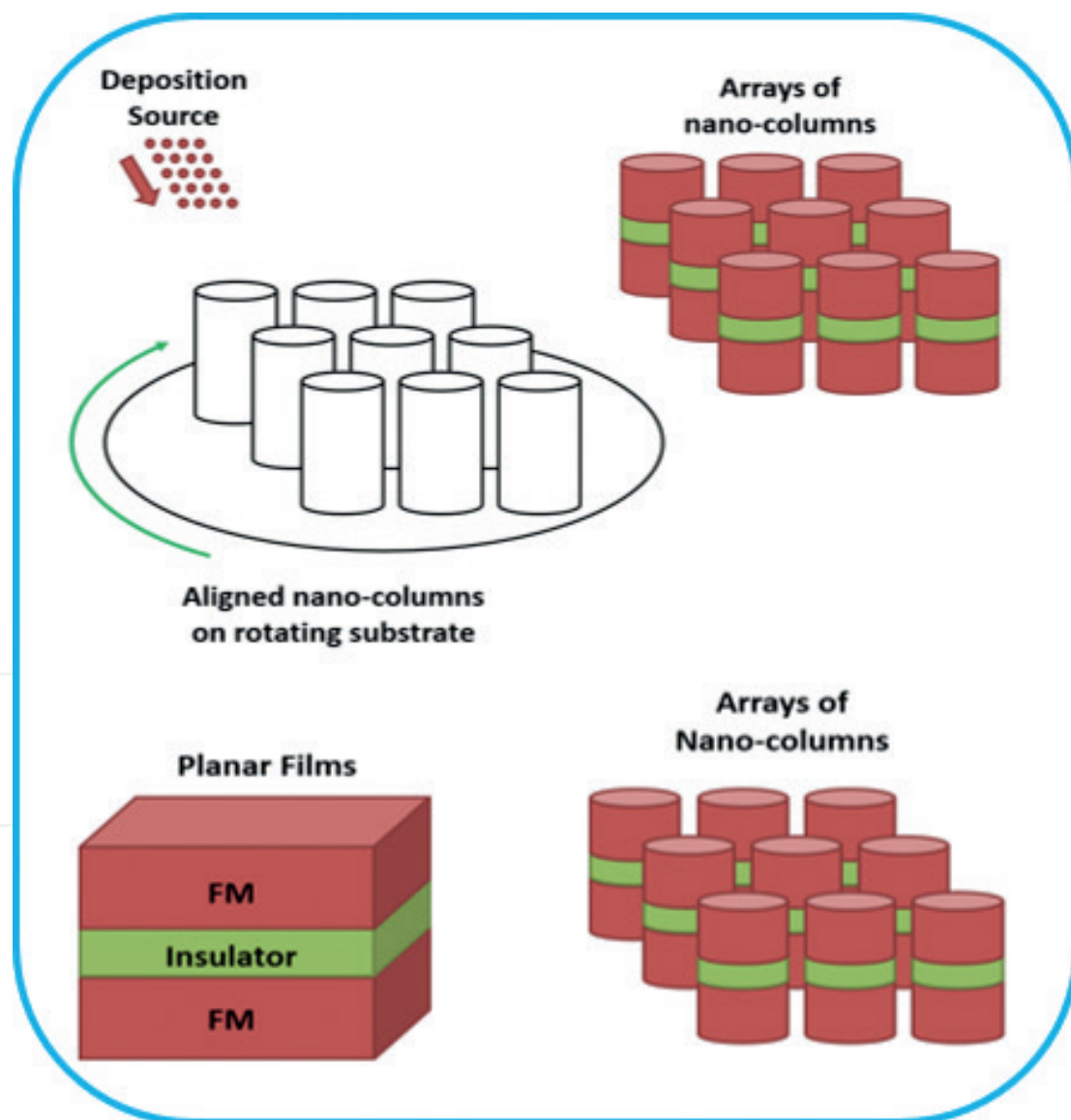


Figure 2. Schematic of planar TMR and array of TMR structures. Both sets of samples were grown using the same magnetron sputtering deposition tool. The planar films will establish initial growth conditions for arrays of nanocolumns.

anisotropy sufficient to overcome the demagnetizing field. This feature makes it important for high-density magnetic media, highly sensitive sensors and parts for a quantum computer. This system in addition to its enormous potential for technological applications is an attractive research object in nanomagnetism.

Samples were prepared in the form of nanometric thin films and nanocolumns as shown in **Figure 2**. Nanocolumn arrays were fabricated with different shapes and in-plane orientations by glancing angle deposition (GLAD). This will provide shape anisotropy, which will compete with surface and volume anisotropy. Many unique and fascinating properties have already been demonstrated by nanocolumns synthesized using GLAD, such as superior mechanical toughness, higher luminescence efficiency, enhancement of thermoelectric figure of merit and lowered lasing threshold. Homogeneous nanowires and nanowire networks have been previously used as chemical sensors, field-effect transistors and inverters, photodetectors, light-emitting diodes, lasers and logic gates. Very recently, by altering the compositions of the nanostructures during fabrication, super-lattice nanowire has been demonstrated, which can greatly increase the versatility and application of these building blocks in nanoscale electronic, photonic, and biological applications. Fe nanocolumns were used to synthesize metal-assisted protein crystallization [10]. Possible applications, including thermoelectrics, nanobarcodes, injection lasers and one-dimensional waveguides, could be implemented through these super-lattice nanostructure building blocks. One very important issue associated with these studies is how to assemble one-dimensional nanostructures in an effective and controllable way. GLAD produces columnar structures through the effect of shadowing during film growth, while the substrate rotation controls the shape of the columns.

2. Experiment

Nanowires, planar nanometric thin films and nanocolumns of Fe/MgO/Fe were synthesized using magnetron DC/RF sputtering, AXXIS sputtering tool from Kurt J. Lesker Company. Fe target of purity 99.9% and MgO of purity 99.95% were sputtered using high-purity Ar. Nanowires were synthesized by magnetron DC and RF sputtering in the interior cylindrical space of carbon nanotubes by magnetron DC and RF sputtering.

Thermal chemical vapor deposition method (TCVD) was used to grow vertically aligned CNTs on the SiO₂ substrate thus filled with Fe/MgO/Fe in this experiment [11]. TCVD involves exposing silica structures to a mixture of ferrocene and xylene at 770°C for 600 s. In TCVD, the furnace is pumped down to about 200 mTorr in an argon bleed, and then, the temperature is raised to 770°C. The solution of ferrocene dissolved in xylene of concentration about 0.01 g/ml and was preheated in a bubbler to 175°C and then passed through the tube furnace. The furnace is cooled down to room temperature, and tips of the CNTs were exposed to plasma to open the nanotubes for the next procedure. The open-ended CNT tips were filled with Fe/MgO/Fe using DC magnetron for the Fe and RF sputtering method for the insulator (MgO) at a substrate temperature of 100°C. This substrate temperature, 100°C, yielded in planar samples the highest value of coercive field compared to several other synthesis substrate temperatures [12].

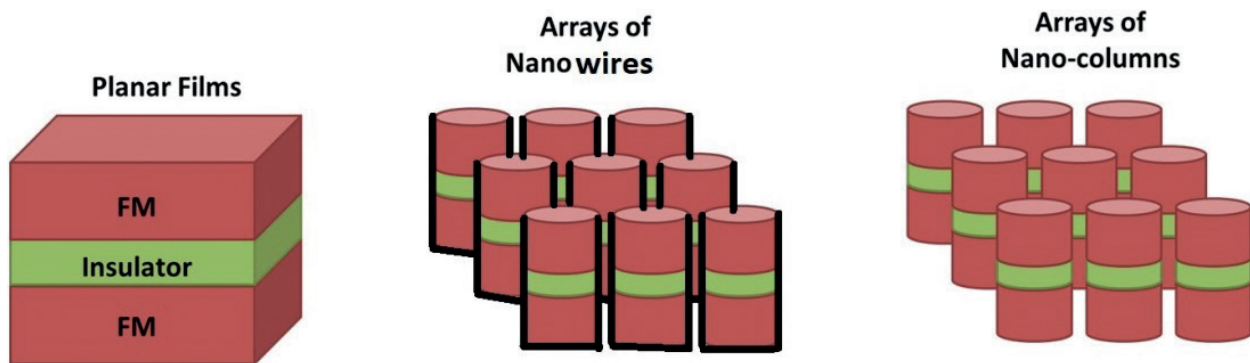


Figure 3. Schematic of planar TMR and array of TMR structure.

Nanowires were synthesized by magnetron DC and RF sputtering in the nanometric interior cylindrical volume of carbon nanotubes at a substrate temperature of 100°C.

Nanometric thin films were epitaxially grown on the MgO (100) substrate of dimensions 5 mm × 5 mm × 0.5 mm using magnetron DC and RF sputtering at several temperatures. All substrates were degassed at 350°C in vacuum of 0.1 μTorr for 1800 s, and samples were pre- and post-annealed at a preselected deposition temperature for 1800 s in vacuum. The source substrate distance was kept fixed at 30 cm, and the substrate surface normal was kept at 45° with a line connecting the center of the sample to the center of the target, while being rotated at a constant rate of 20 rpm for uniform deposition. Under these conditions, epitaxial Fe grows on MgO (100) due to a good lattice match of MgO and Fe, and weak interface interaction [13, 14] free standing Fe is formed. The deposition rate for Fe was 0.17 nm/s as calibrated by the deposition time versus thickness measurements for Fe films several hundred nm thick. In my previous research on thin films of Fe/MgO/Fe, several planar samples were synthesized at several substrate temperatures [12]. The film synthesized at 100°C has the highest saturation magnetization.

Nanocolumns of Fe/MgO/Fe were synthesized at a glancing angle of 70°, and other parameters remained the same as in the synthesis of thin film (**Figure 3**).

All sets of samples were grown using the same magnetron sputtering deposition tool. The planar films established initial growth conditions for the arrays of nanowires and nanocolumns.

3. Structural characterization

Thin film samples of Fe/MgO/Fe on MgO (100) substrates were characterized by XRD (miniflex Rigaku X-ray diffraction of 40 kV/40 mA) using CuKα radiation in θ–2θ geometry (**Figure 4**).

Surface morphologies of nanowires of Fe/MgO/Fe grown in the interior cylindrical space of CNTs were characterized by SEM/STEM in a previous study [12]. The SEM/STEM scan depicts a uniform composition.

X-ray absorption spectroscopy (XAS) measurements on thin films and nanowires of Fe/MgO/Fe carried out at beamline 4UB at the National Synchrotron Light Source (NSLS) in

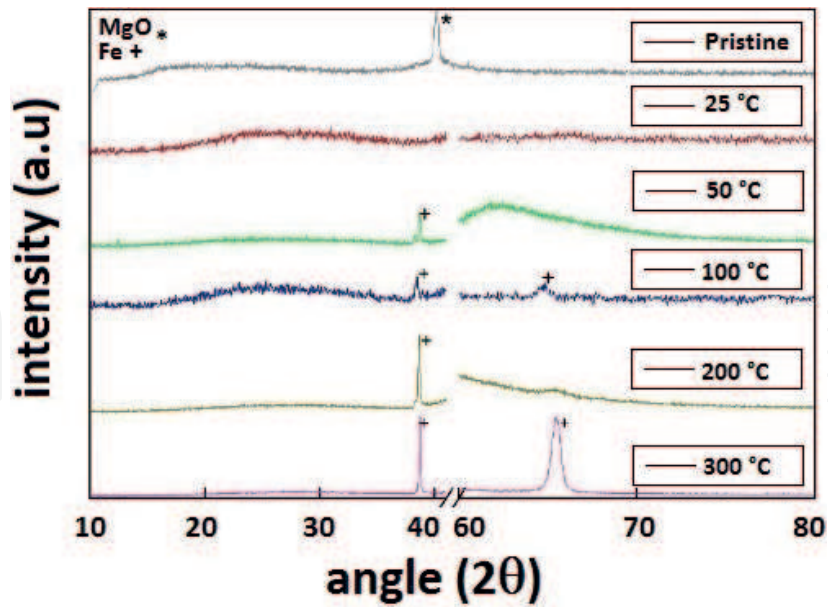


Figure 4. XRD of Fe/MgO/Fe/MgO (100) synthesized at several substrate temperatures and of pristine MgO (100).

Brookhaven National Laboratory (BNL) by the author revealed the existence of a missing shoulder at the Fe L_3 edge in the nanowire spectrum at 717 eV. At the Fe L_2 edge, the double peaks at 723.5 eV are of equal height for the nanowires and a leading peak in the film's spectra.

4. Results from magnetic measurements

In a previous work, it was shown that magnetic force microscopy (MFM) of nanowires of Fe/MgO/Fe revealed the presence of stripe domains [12]; stripe domain was totally absent in films, and it was slightly present in nanocolumns. This shows that the moments are completely aligned in plane in case of thin films. In nanowires, the magnetic moment has a significant out-of-plane component.

X-ray magnetic circular dichroism (XMCD) measurements of nanowires of Fe/MgO/Fe were carried out at beamline 4UB at NSLS in BNL [12]. Background-corrected XMCD signal shows that the nanowires' XMCD signal at the Fe L_3 and L_2 edges is larger [12]. Moreover, a switching between the two edges occurs, and at around 712 eV of photon energy, the intensity of the nanowires' XMCD is smaller [12].

Vibrating sample magnetometer (VSM) measurements were carried out using Vector Magnetometer Model 10 VSM system from MicroSense equipped with 3 T electromagnet (Figures 5–7). In these figures, the coercive field, saturation and remanent magnetization values of thin films, nanowires and nanocolumns of Fe/MgO/Fe synthesized at the substrate temperature of 100 °C versus the angle between the applied magnetic field and the surface normal are shown.

In **Figure 5**, the coercive field (H_c) of nanocolumns (NC), nanowires (NW) and thin films (TF) of Fe/MgO/Fe at several angles between the applied field and the sample surface varying

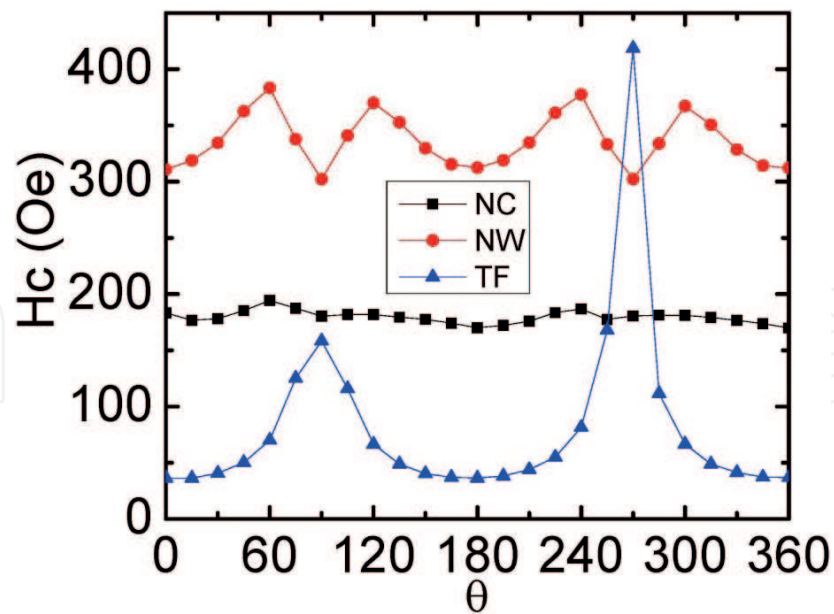


Figure 5. Coercive field (H_c) of nanocolumns (NC), nanowires (NW) and thin films (TF) of Fe/MgO/Fe at several angles between applied field and the sample surface varied between 0 and 360°.

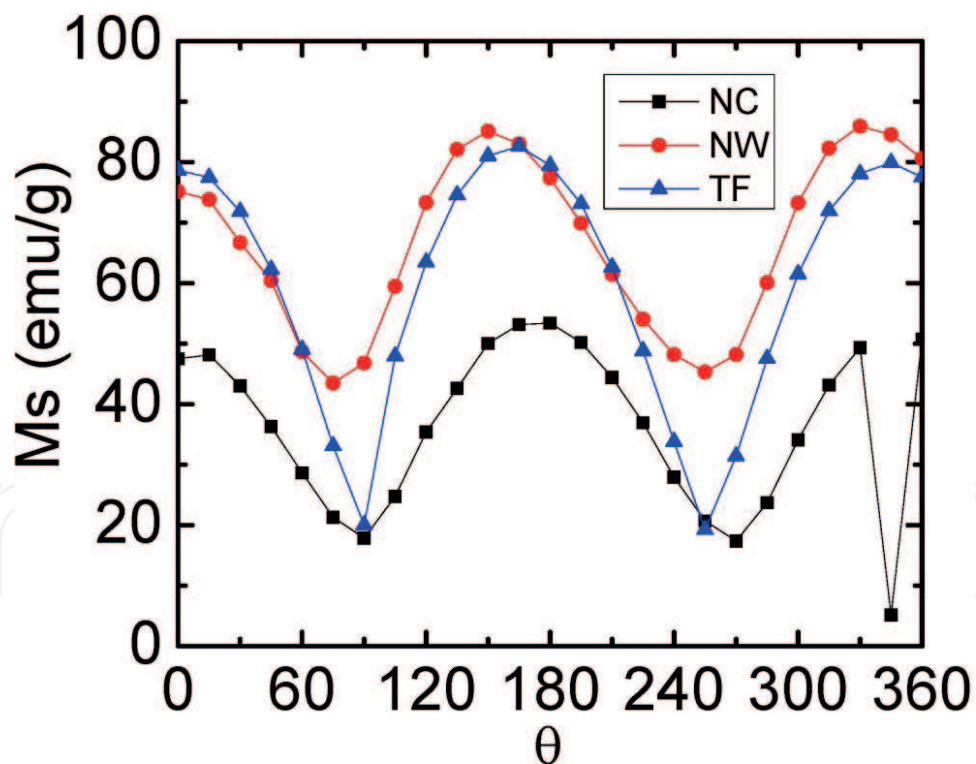


Figure 6. Saturation magnetization (M_s) of nanocolumns (NC), nanowires (NW) and thin films (TF) of Fe/MgO/Fe at several angles between applied field and the sample surface varied between 0 and 360°.

between 0 and 360° is depicted. The coercive field value of nanowires is higher than both nanocolumns and thin films. At an angle of 270°, there is an outlier value for thin films that is higher than both nanowires and nanocolumns value of H_c at 270°. The H_c values of nanowires

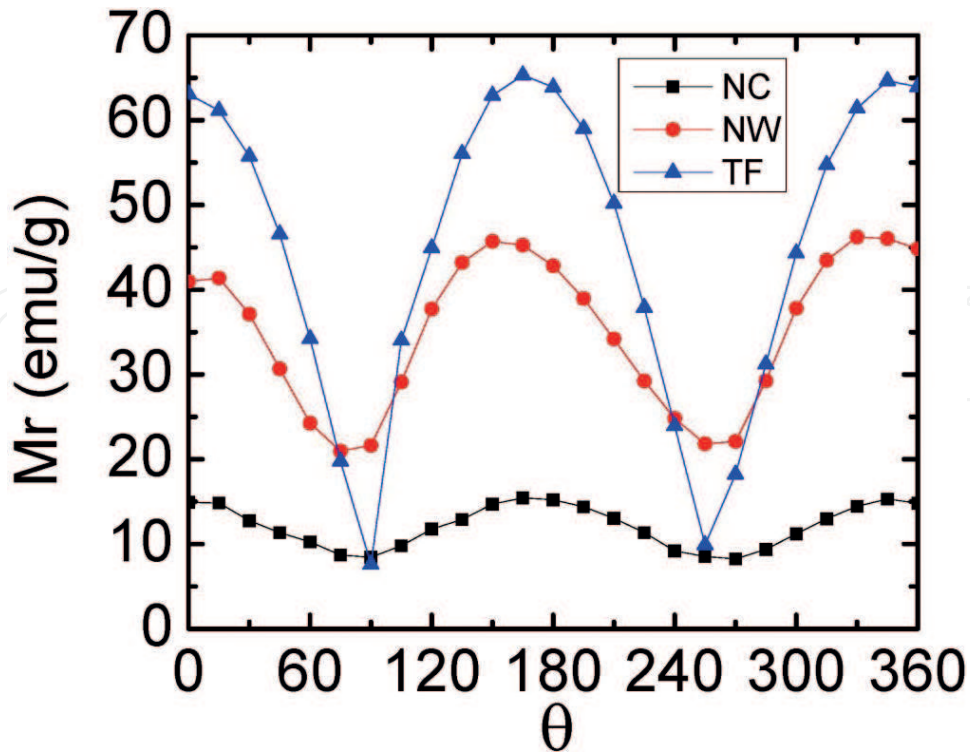


Figure 7. Remanent magnetization (M_r) of nanocolumns (NC), nanowires (NW) and thin films (TF) of Fe/MgO/Fe at several angles between applied field and the sample surface varied between 0 and 360°.

oscillate with a period of 90°, whereas those of thin films oscillate with a period of 180°. For nanocolumns, the coercive field values are constant at all angles. The in-plane value of the coercive field of nanowires is higher by 754% than planar thin films.

Saturation magnetization (M_s) of nanocolumns (NC), nanowires (NW) and thin films (TF) of Fe/MgO/Fe at several angles between the applied field and the sample surface varying between 0 and 360° is depicted in **Figure 6**. The saturation magnetization of all three oscillates with a period of 180°. The saturation magnetization of nanowires is slightly higher than nanocolumns and thin films at all angles.

Remanent magnetization (M_r) of nanocolumns (NC), nanowires (NW) and thin films (TF) of Fe/MgO/Fe at several angles between the applied field and the sample surface varying between 0 and 360° is shown in **Figure 7**. The remanent magnetization of all three oscillates with a period of 180°. The remanent magnetization of thin films is higher than nanowires and nanocolumns at all angles except at 90 and 270°.

Magnetic torque measurements for nanocolumns of Fe/MgO/Fe synthesized at 100°C for several applied fields were carried out using the EV7 torque magnetometer (TMM) system equipped with a 2 T electromagnet (**Figure 8**). Torque magnetometer measurements of thin films and nanowires results from the previous study show a similar trend such as a twofold symmetry [12]. TMM measurements of nanocolumns of Fe/MgO/Fe synthesized at 100°C for several applied fields are shown in **Figure 8(a)** and in the polar plot in **Figure 8(b)**. TMM measurements for planar film and nanowires of Fe/MgO/Fe both synthesized at 100°C were

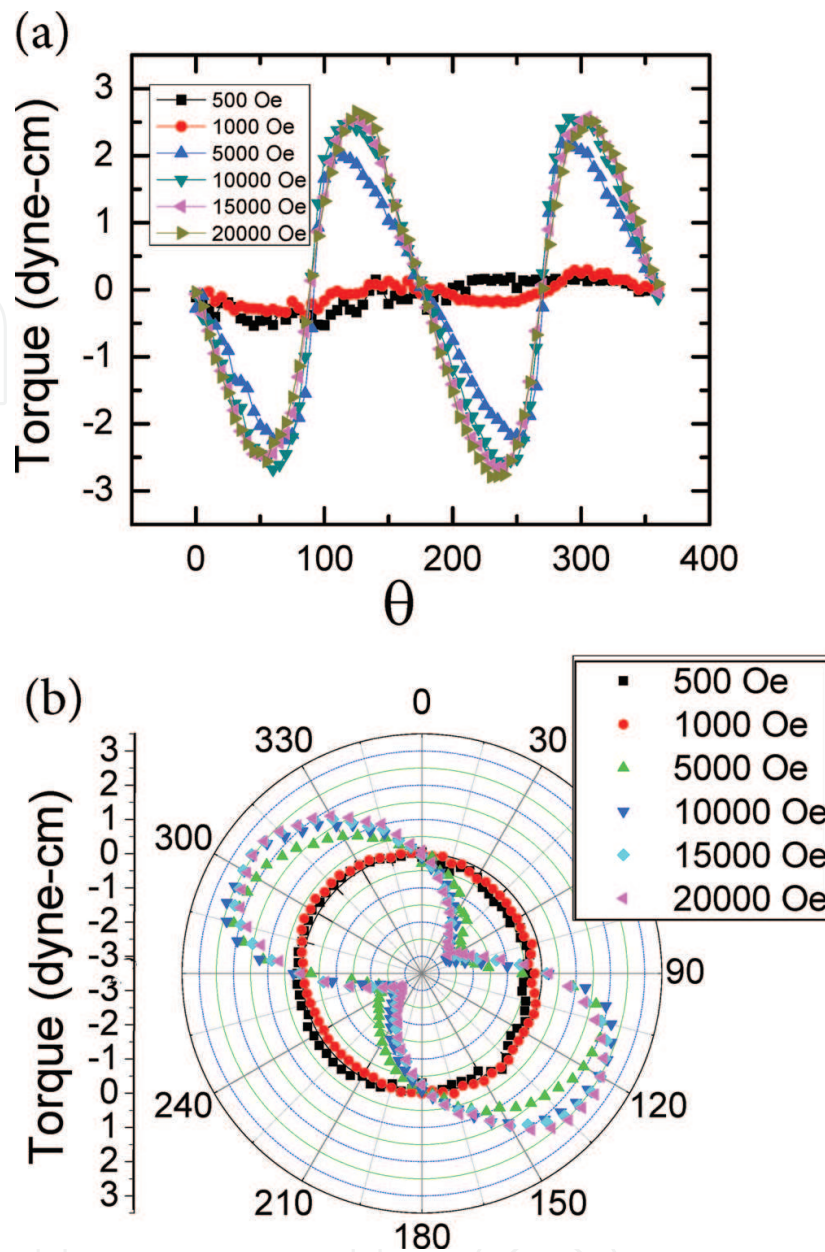


Figure 8. (a) TMM measurements of nanocolumns of Fe/MgO/Fe at several fields while the angle between applied field and the sample surface rotate between 0 and 360°. (b) The same figure in part a in polar plot.

shown to have pronounced twofold symmetry in the rotational hysteresis loop that appeared for an applied field of 5000 Oe and higher [12]. Here, it is shown for the first time that nanocolumns also show pronounced twofold symmetry in the rotational hysteresis loop for an applied field of 5000 Oe and higher (**Figure 8**). Torque on the film is 20 times higher than on nanowires for the same applied field [12]. **Figure 8** shows that TMM measurements for nanocolumns in the polar plot along equivalent crystallographic direction yields torque on the nanocolumns are 10 times higher than on nanowires for the same applied field.

Figure 9 depicts M_r/M_s ratio measured using VSM at several angles with respect to an applied field for thin films, nanowires and nanocolumns of Fe/MgO/Fe synthesized at 100°C. External

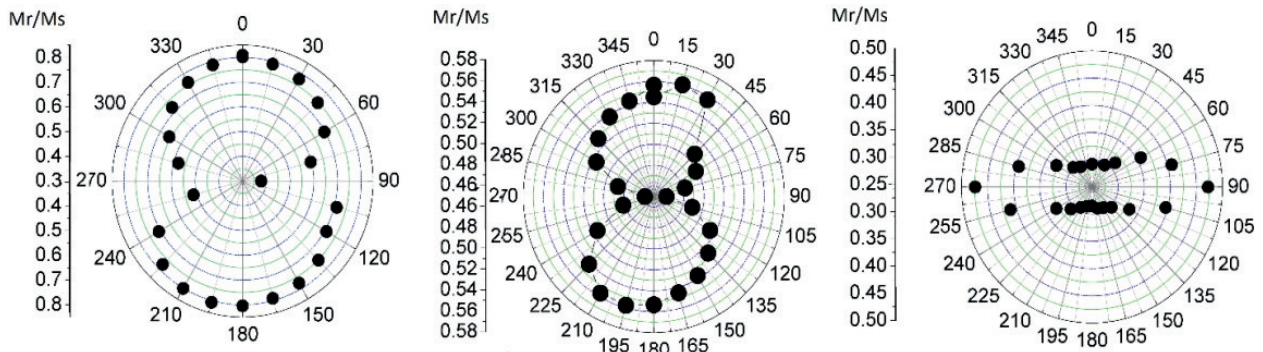


Figure 9. (a) Mr/Ms measurements of thin films Fe/MgO/Fe at several fields. (b) Mr/Ms measurements of nanowires of Fe/MgO/Fe at several fields. (c) Mr/Ms measurements of nanocolumns of Fe/MgO/Fe at several fields.

magnetic field applied along equivalent crystallographic directions did not produce equivalent hysteresis loops. This is obvious when comparing Mr/Ms ratio obtained for symmetric orientations, every 15° from 0 to 360° as shown in **Figure 9**. Mr/Ms ratio extracted from VSM measurements at every 15° between 0 and 360° for planar film, nanowires and nanocolumns of Fe/MgO/Fe synthesized at 100°C is shown in **Figure 9**. In all three cases, a twofold symmetry is observed.

Figure 10 depicts a coercive field measured using VSM at several angles with respect to an applied field for thin films, nanowires and nanocolumns of Fe/MgO/Fe synthesized at 100°C. **Figure 10** depicts a coercive field measured using VSM at several angles with respect to an applied field for thin films, nanowires and nanocolumns of Fe/MgO/Fe synthesized at 100°C. A strong dependence on an angle was observed in the films than in the nanowires. Coercive field (Hc) value extracted from VSM measurements at every 15° between 0 and 360° for planar films, nanowires and nanocolumns of Fe/MgO/Fe synthesized at 100°C is shown in **Figure 10**. In all three cases, different symmetries are observed. Nanowires depict a fourfold symmetry, whereas a well-defined twofold symmetry is observed in nanocolumns. The conspicuous symmetry difference in nanowires could be due to shape anisotropy and also due to the hybridization that occurs between the π -electronic states of carbon and 3d bands of the Fe surface.

Figure 11 depicts temperature-dependent magnetization measurements of planar nanometric thin films of Fe/MgO/Fe/MgO (100) synthesized at 100°C. The planar thin film with the highest

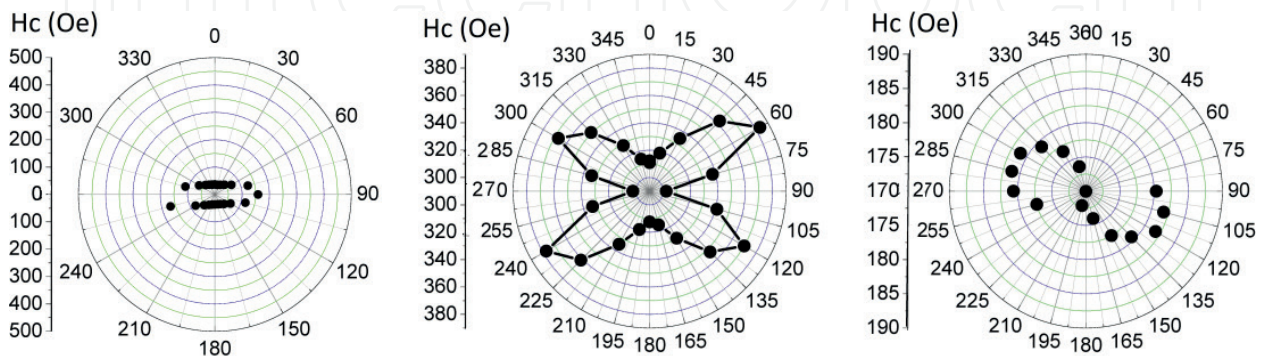


Figure 10. (a) Hc measurements of thin films of Fe/MgO/Fe at several fields. (b) Hc measurements of nanowires of Fe/MgO/Fe at several fields. (c) Hc measurements of nanocolumns of Fe/MgO/Fe at several fields.

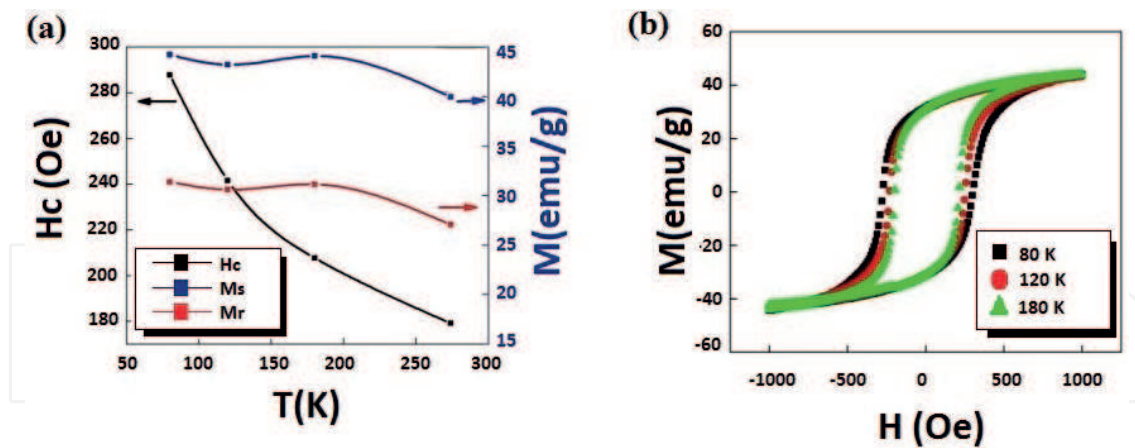


Figure 11. (a) Low-temperature VSM measurements of H_c , M_s and M_r measured at several temperatures for thin films of Fe/MgO/Fe synthesized at 100°C. The lines are drawn connecting the points as a guide. (b) Hysteresis loops of Fe/MgO/Fe/MgO (100) thin film synthesized at 100°C measured at several low-temperature values.

value of H_c as shown in **Figure 11(a)** is the sample synthesized at 100°C. Low-temperature VSM measurements of thin films of Fe/MgO/Fe synthesized at 100°C are shown in **Figure 11**; as expected, all three magnetization parameters H_c , M_r , and M_s increased as temperature is lowered; more pronounced increase was observed in H_c . As shown in **Figure 11(a)**, the value of H_c increased by 61%, M_r increased by 16% and M_s increased by 11% at 80 K compared to their room temperature values, respectively. **Figure 11(b)** depicts hysteresis loops of the Fe/MgO/Fe planar film at low temperatures.

5. Discussion

As shown in **Figure 5**, the in-plane coercive field (H_c) of nanowires is higher than thin films' H_c by 754%, and nanocolumns' H_c is higher than thin films' H_c by 403%. These higher values of H_c for nanowires are due to magneto crystalline shape anisotropy since both nanowires' and nanocolumns' H_c values are higher than H_c of planar films. Magneto crystalline anisotropy according to density functional theory is due to change in the relative occupancy of the 3d orbitals of Fe atoms at the interface of Fe/MgO [15]. The reason nanowires' H_c is higher than nanocolumns' H_c is encapsulation by carbon nanotubes and subsequent interaction of C atoms with Fe atoms. This interaction between C and Fe atoms is between the π -electronic states of carbon and 3d bands of the Fe surface. First-principle calculation predicts weak interaction between the Fe layer and the MgO substrate making the Fe film to act as a free-standing Fe monolayer ($3.10 \mu_B$), with enhanced magnetic moment [13]. Mössbauer measurement has also shown higher hyperfine field attributed to the interface region between the epitaxial Fe and MgO layers [16]. This study on the hybrid interface between carbon-based organic molecules and ferromagnetic surfaces is very important in the development of wearable spintronics and environmentally friendly sensors based on organic spintronics [17]. Nanowires of α -Fe synthesized inside alumina templates and single nanowires inside dense nickel nanowire arrays [18, 19] depict a B-H loop that narrows when the field is perpendicular

to the nanowire axis; this deformation in the B-H loop indicates small dipole interactions. The small dipole interactions in these systems are due to the large spacing between the nanowires. In this chapter, nanowires of Fe/MgO/Fe for all orientations of the applied field depict B-H loops that have the same width without narrowing deformation in the B-H loops indicating high dipole interactions. The reason for this high dipolar interactions is the densely grown nanowires with close proximity to each other.

6. Conclusions

In conclusion, this chapter reports on the magnetic study of Fe/MgO/Fe of planar nanometric thin films, nanocolumns and nanowires encapsulated inside carbon nanotubes. Deposition conditions in particular geometry and most notably encapsulation influence both structural and magnetic properties of Fe/MgO/Fe TMR. Nanowires of Fe/MgO/Fe showed superior magnetic properties than planar thin films. The in-plane value of the coercive field of nanowires is higher by 754% than planar thin films.

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