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Magnetic, electronic structure and interface study of Fe/MgO/Fe multilayer

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ABSTRACT

MgO based magnetic tunnel junctions (MTJs) exhibit high tunneling magnetoresistance (TMR) and have potential applications in magnetic random access memories. This study addresses the role of interface in the Fe/MgO/Fe based MTJs. For present investigation, Fe/MgO/Fe multilayer stack on Si substrates is grown by electron beam evaporation method and has been investigated for structural, magnetic and electronic properties. All the layers in the stack were of polycrystalline in nature as evidenced from X-ray diffraction studies, and the magnetic measurements show the attributes perpendicular magnetic anisotropy. Results from near edge X-ray absorption spectra at Fe L-edges measured by total electron yield mode and X-ray reflectometry indicate the formation of FeO_x at the Fe/MgO interface. These are associated with hybridization of Fe (3*d*)-O(2*p*) levels at Fe/MgO interface in the stack and thickness of layers in the stacks. Absence of magnetic de-coupling between top and bottom ferromagnetic layers has been attributed to interface roughness and oxidation at Fe/MgO interface. This study highlights the role of interface and oxidation that need to be considered for improving the TMR for devices.

Keywords: Magnetic multilayers; e-beam evaporation; X-ray absorption spectroscopy; X-ray reflectometry.

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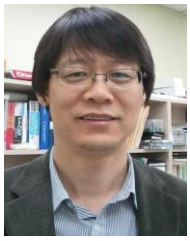

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Introduction

Magnetic tunnel junctions (MTJs) with crystalline MgO barrier are subject of great interest due to their high tunnel magnetoresistance (TMR) and their applications in magnetic random access memories (MRAM) [**1, 2**]. These devices exhibit various phenomena like Seebeck effect [3], thermopower [**4**] and theoretically predicted spin caloritronic [**5**] and thereby have become focus of attention

for scientific community. It has been shown by theoretical calculations [**6, 7**] that these devices show high TMR up to ~1000% with Fe/MgO/Fe layers in MTJs. Experimentally, MgO-based MTJs with Fe electrode fabricated by various groups have exhibited TMR ratios as high as over 200% at room temperature [**8, 9**] and increase over to 604% when CoFeB has been used as a ferromagnetic electrodes. Such a discrepancy between theoretical and experimental values of TMR is associated with the interface properties of the heterostructures, as spin dependent tunneling is sensitive to the interface [**10**]. Hence, the growth and investigation of interfaces properties of Fe/MgO/Fe becomes important for improving the device performance of MTJs. Many groups have simulated the interface between Fe/MgO by using first principle calculations [**11, 12**]. Oxygen induced symmetrisation and structural coherency in these junctions lead to increment in TMR value. The formation of FeO at the interfaces increases the structural coherency, giving rise to higher values of TMR ratio [**13**]. Role of various interfaces on the properties of Fe/MgO/Fe based MTJs have been investigated theoretically and found that TMR depends on the interface roughness [**14**]. Spectroscopic techniques using third generation synchrotron radiation sources have emerged as powerful tools to understand the chemical changes occurring at the interface in multilayers. This technique provides the information of the magnetic moments of magnetic layers of Fe/MgO/Fe and Co_2MnSi ultrathin films facing an epitaxial MgO(001) tunnel barrier MTJs [**15, 16**] and to explain the observed B diffusion and crystallization of CoFeB layers within a few seconds of the post growth high temperature annealing for CoFeB/MgO/CoFeB MTJs. This technique also help to understand the increase in structural order with annealing that gives rise to increase in TMR [**17, 20**]. In case of MTJs, the efforts have been made to improve the interface roughness and crystalline quality of MgO barrier by introducing buffer or underlayer and annealing the whole structure in ultra-high vacuum [**21-23**]. Besides the post deposition treatment, deposition method also plays an important role in determining the properties of MTJs. Although methods like molecular beam epitaxy (MBE) and rf-sputtering are employed for fabricating the multilayers stack of MTJs and are most popular but e-beam evaporation method is also preferred to fabricate MgO layer of the multilayer stack as this method provides layer by layer growth and MTJs in which MgO barrier is grown by this method exhibit reduced magnetic noise [**24**]. In addition, this method is cost effective.

Present study focuses the fabrication of Fe/MgO/Fe multilayer stack on Si substrate by e-beam evaporation and the structural, magnetic, electronic and interface characterization. Magnetic properties were studied by using vibrating sample magnetometer (VSM) and local electronic structure by the near-edge X-ray absorption fine structure (NEXAFS) spectroscopic technique. To understand the interface, X-ray reflectivity (XRR) measurement was carried out. Above results bring out the role of interfaces and oxidization of interface in the properties of this multilayer stack.

Experimental

Materials and methods

For deposition, high purity MgO, Fe and Au (99.99%) materials have been procured from *Alfa Aesar and Sigma Aldrich*. Multilayer stack of Fe/MgO/Fe/Au on Si(100) substrate was fabricated by e-beam evaporation method with base pressure better than 5×10^{-8} Torr. First, a 10 nm MgO buffer layer was deposited on Si-substrate in order to prevent silicide formation at Si/Fe interface due to diffusion, also MgO works as good under layer to grow Fe epitaxially [**6, 7, 10**]. The substrate temperature was kept at 300 °C during deposition. On top of MgO buffer layer a 20 nm Fe thin film was deposited at 180 °C. Subsequently, MgO barrier layer of 5 nm thickness was deposited at the same growth temperature. The upper Fe-layer of 10 nm thickness was deposited at temperature of 200 °C. This multilayer stack was annealed at 315 °C for 1.5 hr in ultrahigh vacuum to improve crystallinity and to have sharp interface. After annealing, a 10 nm Au capping layer was deposited at room temperature in order to prevent oxidation of Fe [**25, 26**].

Characterization techniques

Rutherford backscattering spectrometry (RBS) studies for these multilayer structures were performed at Inter University Accelerator Centre, New Delhi and results obtained from RBS study have been published elsewhere **[26]**. X-ray diffraction (XRD) studies on these stacks were carried out on a Philips X-ray diffractometer at Indian Institute of Technology, New Delhi. Magnetic studies on these samples were carried out on a VSM at National Physical Laboratory, New Delhi. In order to understand the electronic structure at interface as well as Fe-layer NEXAFS technique has been utilized. NEXAFS spectra were recorded at the high energy spherical grating monochromator (HSGM) BL20A1 **[27]** beamline in the National Synchrotron Radiation Research Center (NSRRC), Hsinchu, Taiwan in total electron yield (TEY, surface sensitive) and total fluorescence yield (TFY, bulk sensitive) modes. XRR measurements were also carried out for structure and the results obtained were simulated by using X'pert reflectivity software.

Results and discussion

Structural and magnetic studies

As evident from **Fig. 1**, the XRD pattern exhibits polycrystalline nature of various layers in the multilayer stack. **Fig. 2** shows the hysteresis curves of this stack while applying the magnetic field parallel and perpendicular to the film surface. Both these curves are asymmetric with respect to applied field and magnetic moment axis. Hysteresis loop in parallel direction is shifted by 45 Oe

from zero. The coercivity of this loop is 63 Oe and remanence is 44 μemu. The positive and negative saturation magnetic moments of this film in parallel direction are respectively 368 and 232 μemu. The coercivity value for perpendicular hysteresis is 89 Oe and the remanence is 169 μemu. Apart from hysteresis in parallel direction, the magnetic moments does not saturate up to 5000 Oe, however, a square hysteresis appears in the range -200 to 200 Oe. Presence of shifted hysteresis loops in both the directions may be due to presence of exchange bias. The origin of these phenomena may be due to oxidation of Fe at the top interface surface, which may cause ferrimagnetic γ-Fe_2O_3 at Fe/MgO interface. However, positive vertical shift along magnetization axis may be due to the pinned interfacial uncompensated spins of ferrimagnetic γ-Fe_2O_3 at the interface [**28**]. Phenomena of exchange bias may be attributed to ferromagnetic coupling between uneven number of spins at the interface of formed Fe-oxide layer and antiferromagnetic coupling at the interface, however presence of defects which will have the spins along the applied field, providing uncompensated spins, causes vertical shift in hysteresis curve. Absence of steps in the hysteresis of stack may be attributed to the cumulative effects of oxidation of Fe at the top interface surface and discontinuous nature of MgO films or the rough interface.

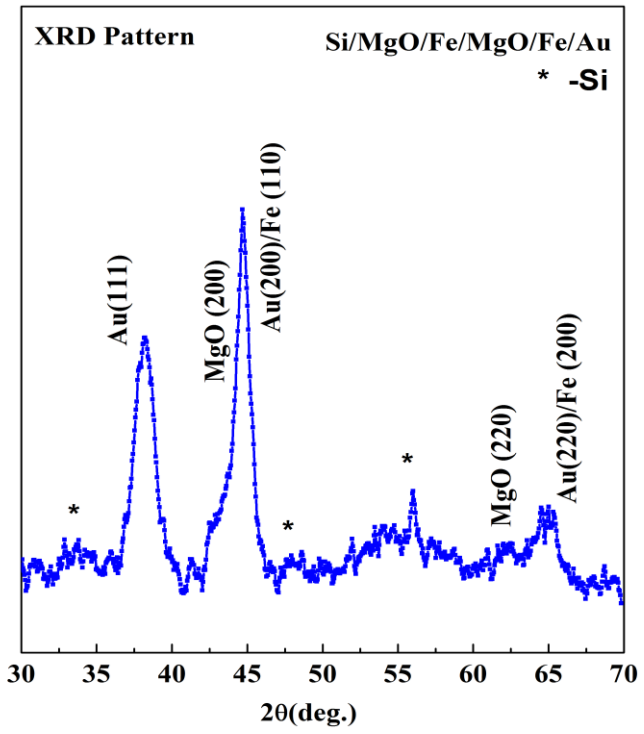

Fig. 1. X-ray diffraction pattern of Fe/MgO/Fe multilayer stack.

It is known that when film is deposited thin enough, it can form magnetic islands or clusters rather than a continuous film. For these magnetic clusters the ambient thermal energy is sufficient to cause relaxation of the magnetization vectors of the clusters, leading to small value of coercivity. S-shape behavior of hysteresis in parallel direction is an indication of this behavior [**29**]. The non-saturation and square like shape in perpendicular direction also favors this behavior. The square like shape at low field in the perpendicular hysteresis of this stack may be due to presence of perpendicular magnetic anisotropy (PMA), however, non-saturation of hysteresis in this direction arises due to small magnetic clusters. The PMA which is generally arises due to Fe (3*d*) and O (2*p*) hybridization at metal-oxide layer interface is occurring in the stack [**30**]. Observation of a small signature of PMA is an important result in this work and generally not observed in thick MTJ stack. Most important assumption used in discussing the VSM results is oxidation at Fe/MgO interface; hence NEXAFS spectra of these stacks have been recorded at Fe L-, and Mg, O K-, edges.

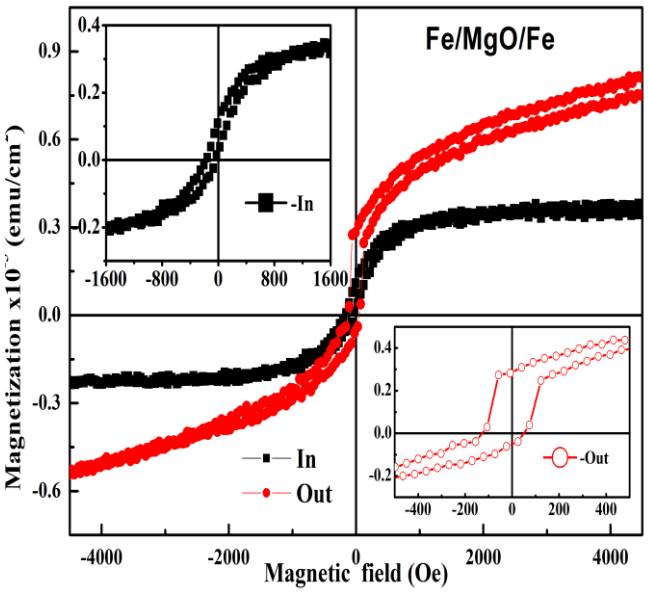

Fig. 2. Hysteresis curves of Fe/MgO/Fe in parallel (in) and perpendicular (out) direction. Inset in upper left half shows in plane curve at low field, however at lower right half it shows perpendicular curve at low field. A squared feature is evident at low field in perpendicular hysteresis.

NEXAFS study

Fig. 3 shows the Fe L-edges spectra for the multilayer measured in TEY and TFY modes. NEXAFS spectra recorded for Fe L-edges in TEY mode show the spectral features at 708, 710, 721 and 723 eV. These arise due to excitation of Fe (2*p*) core level to the 3*d* empty states. Due to spin-orbit coupling, it gives rise to degenerate states $2p_{3/2}$ and $2p_{1/2}$ showing multiplets centered at 709 and 712 eV (**Fig. 2**). The octahedral crystal fields lifts the degeneracy of the $2p_{3/2}$ and $2p_{1/2}$ levels so that two levels with t_{2g} and e_g symmetry are created, as indicated by the two structures at about 708 and 710 eV and at 721 and 723 eV in the spectra (**Fig. 2**). These features are indicative of Fe^{3+} oxidation state in all the samples and generally observed in Fe-based oxide systems [**31**]. Since, TEY mode is surface sensitive, it may be contemplated that MgO/Fe interfaces in both the multilayers have oxidized Fe at interface. The oxidation of upper Fe-layer is not expected in the present case because this layer is capped on the top by Au-layer.

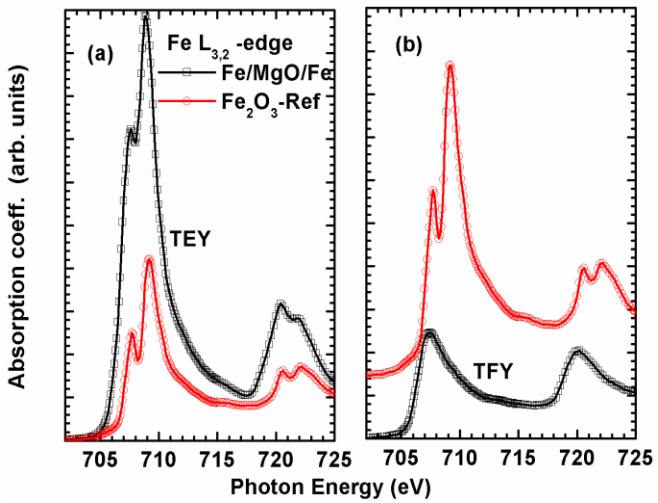

Fig. 3. Normalized Fe $L_{3,2}$-edge spectra for Fe/MgO/Fe multilayer stack in (a) TEY and (b) TFY modes with Fe_2O_3 as reference. (a) Spectral features taken in TEY is similar to Fe_2O_3, showing Fe-oxidation at Fe/MgO and MgO/Fe interface, however (b) bulk sensitive mode (TFY) exhibits different features than the reference sample.

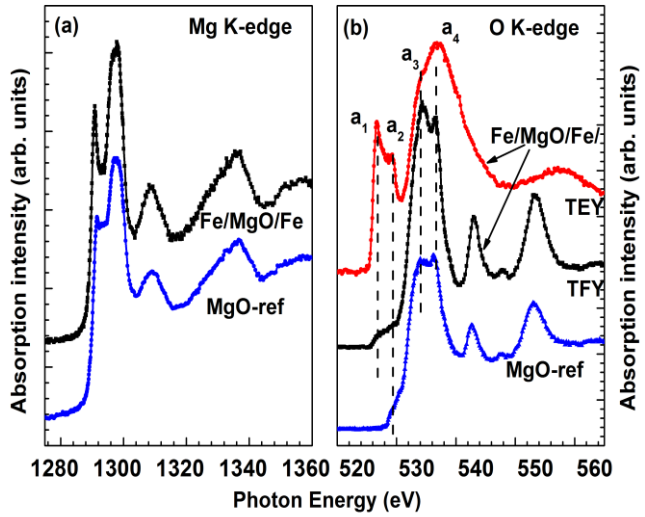

Fig. 4. Normalized (a) Mg-K-edge spectra for Fe/MgO/Fe multilayer (black line), red line spectrum corresponds to the reference sample, (b) O K-edge spectra for Fe/MgO/Fe multilayer taken in TEY and TFY modes. Spectral features in TEY mode is surface sensitive mode and different from those of TFY mode (bulk sensitive) showing the difference in the electronic structure (corresponding to O(2*p*)-Fe(3*d*) hybridization) at interface.

The NEXAFS spectra of Fe L-edges for multilayer were recorded in TFY mode and exhibit peaks at 708 and 714 eV (**Fig. 3**). This indicates that Fe ions are not bonded with oxygen or any other atoms in the bulk. This shows the metallic nature of Fe-layer. Hence, Fe-layers are metallic and possibly oxidized at surface attached to MgO. Mg K-edge spectra for the multilayer alongwith reference sample exhibit peaks around the position 1284, 1300, 1308 and 1332 eV (**Fig. 4a**). The presences of these features in the spectra are associated primarily with Mg 3*p*-O 2*p* hybridized states [**32**].

OK-edge spectrum of multilayer in TEY mode consists of pre-edge structure around ~529 eV (**Fig. 4b**). These structures in these films generally due to the excitation to the localized bound state and consistent with the previous reports of NEXAFS study of MgO [**33**]. In this spectrum, the spectral features around 534, 536, 542 and 552.9 eV are observed. These features in the both spectra are very much similar to the spectra of MgO thin film reported by Luchs et al. and arise due to Mg(1*s*)-O(2*p*) hybridized states [**34**]. A visual inspection shows large difference in the TEY and TFY spectra of the samples. This seems to be consistent with the TFY Mg K-edge spectra. Different spectral features observed in the TEY spectrum of the multilayer may be due to different bonding states of oxygen present in the stack. In this spectrum, peaks around ~ 527, 530, 534, 538, 545 eV are observed. These features are very much similar to those of ferrites [**35, 36**], where the spectral features originate from transitions of unoccupied states of O 2*p* character hybridized with 3*d* metal ions. This is in accordance with the formation of Fe-O_x at Fe/MgO and MgO/Fe interfaces. Oxide formation at Fe/MgO or MgO/Fe interface has been theoretically predicted in Fe/MgO/Fe(001) MTJ [**37**] and studied that tunneling magneto-resistance changes on oxygen stoichiometry. This aspect later investigated in Fe/MgO/Fe layer by various methods [**38**], transmission electron microscopy [**39**] and spin dependent tunneling spectroscopy [**40**], which corroborated with our results. Existence of Fe-O_x at Fe/MgO interface has also been supported by X-ray magnetic circular dichroism carried out on this sample **[41]**.

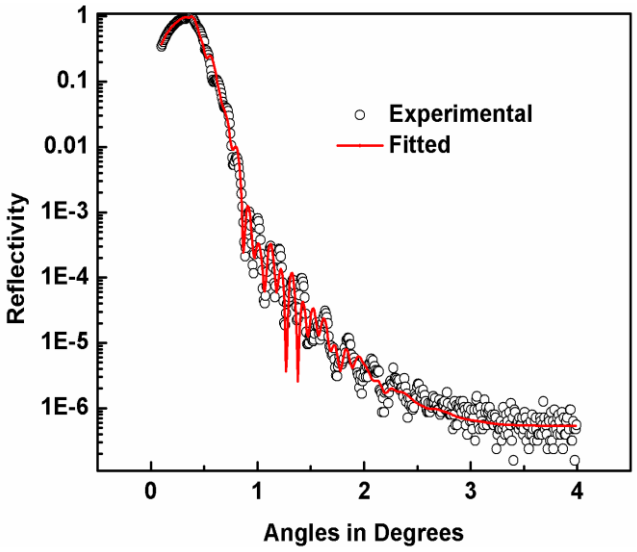

Fig. 5. XRR curve of stack: (a) Experimental (dot circle) and (b) simulated (red line). XRR curve was simulated by considering oxidation of Fe at Fe/MgO and MgO/Fe interface.

XRR study

Results obtained from VSM and NEXAFS imply that the oxidation at Fe/MgO and Fe/MgO interfaces. Further characterization of thickness and interfaces were carried out

by using specular XRR for the multilayer stack. The experimental data (open black circle) is simulated by considering a multilayer model (solid red line) as shown in **Fig. 5**. The simulated parameters viz density of layer (ρ), thickness (t) and interface roughness (σ) are collated in **Table 1**.

Table 1. Density (ρ), Thickness (t) and roughness (σ) of various layers of Fe/MgO/Fe multilayer.

Layers	ρ (gm/cm^3)	t ±0.1 (nm)	σ ±0.01 (nm)
Si	2.328	-	0.15
SiO_x	2.554	2.3	0.82
Mg_2Si_x	0.998	1.0	0.41
MgO	3.456	11.1	1.06
FeO_x	5.054	1.3	1.21
Fe	6.767	12.4	0.68
FeO_x	5.051	1.3	0.69
MgO	3.399	5.7	1.67
FeO_x	4.871	1.9	1.23
Fe	7.306	7.5	1.30
Au	18.778	4.30	2.06

A visual inspection of **Table 1** shows the presence of Fe-oxidation at Fe/MgO and MgO/Fe interfaces in the stack which supports the results obtained from VSM and NEXAFS study. The barrier layer in this stack is ~ 5.7±0.1 nm with interface roughness of ~ 1.67±0.01 nm. Due to large interface roughness of MgO/Fe and Fe/MgO interface, the coupling between upper and lower FM layers is expected as observed in VSM study. Although roughness of MgO is higher, but the density of the grown MgO barrier layer in stacks was found to be 3.39 gm/cc, which is close to the bulk density of MgO (3.58 gm/cc). It is expected that coercivity is also affected by the polycrystalline nature of various layers and oxidation at interfaces inhibiting the presence of magnetic switching between these two layers. Such types of aspects have been discussed by Yuasa *et al.* and correlated with reduced TMR of MTJ [**8**].

Conclusion

Fe/MgO/Fe multilayer stack was deposited by e-beam evaporation method exhibits polycrystalline nature. Magnetic hysteresis loops show absence of characteristics of magnetic tunnel junction stack, however at low magnetic field perpendicular hysteresis is observed and attributed to perpendicular orientation of magnetic moments. The observed magnetic behavior of the stack has been explained on the basis of oxidation of Fe at Fe/MgO and MgO/Fe interfaces, discontinuous nature of MgO and presence of defects. These results from magnetic and NEXAFS studies are very well corroborated with findings from X-ray reflectometry.

Acknowledgements

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