Surface Morphology and Exchange Bias Anisotropy Studies in Large Area Deposited Co₂FeSi/Ir₅₀Mn₅₀ Multi-Layers For Spintronic Applications

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ABSTRACT

Surface morphology and magnetic properties of ferromagnetic Heusler alloy Co_2FeSi thin films and their multi-layers with anti-ferromagnetic $Ir_{50}Mn_{50}$, which find applications in spintronic devices were investigated. The sputtering process flow for large area deposition of thin films on 3 inch size thermally oxidized single crystal Si(100)/SiO₂ substrates have been developed by optimizing the sputtering geometry and other process parameters. A uniform film composition, thickness, smooth surface, good crystallinity and magnetic properties have been achieved in the films over 3-inch size wafers. The isotropic magnetic properties such as saturation/remanent magnetizations, coercivity were achieved in Co_2FeSi films deposited on 3-inch size Si(100)/SiO₂ wafers with 15 nm Cr buffer layer. An exchange bias anisotropy has been established in $Co_2FeSi/IrMn$ multilayer by magnetic annealing process using in-house made magnetic annealing set up. A maximum exchange bias anisotropy field, H_{ex} of 178 Oe and low coercivity, H_c of 85 Oe has been achieved in the $Co_2FeSi/IrMn$ multilayer stacks suitable for magnetic tunnel junctions for spintronic applications.

Keywords: Half metallic ferromagnetism; Multilayers; Magnetic properties; Exchange biasanisotropy

1. INTRODUCTION

Among Heusler alloys, Co, FeSi based alloys have gained much interest due to their high Currie temperature ($T_c \sim 1100$ K) and large magnetic moment (~6 $\mu_{\rm p}/f.u$) along with halfmetallic ferromagnetic character even at temperatures higher than room temperature¹. In half-metallic ferromagnets (HFM), one of the spin sub-bands (generally the majority-spin or upspin sub-band) is metallic, whereas the Fermi level falls into a gap of the other (minority-spin or down-spin) sub-band due to ferromagnetic decoupling. As a result, these materials are 100 % spin polarized at T=0 K and show very large electrical resistivity for one spin direction²⁻³. These Heusler alloys are potential candidate for spin electronic devices such as magnetic sensors, spin transistors, magneto- resistive random access memories and reading heads for hard disk drives because of the high Tunneling Magnetoresistance (TMR) ratio that can be obtained using these alloys as one of the electrodes in a multilayer structure⁴⁻⁶.

Highly sensitive magnetic field sensors are important for detection of magnetic signatures of naval systems such as warships, submarines etc. The stray magnetic field or the magnetic signatures of naval systems need to be minimized to make the systems stealthier. For that purpose, the stray magnetic fields of the naval systems need to be measured accurately using this type of spintronic based sensor for degaussing the naval systems⁷⁻⁸. For uniformity in magnetic properties and economic fabrication/production of the thin film devices, fabrication of magnetic field sensors in large size wafers is advantages. The magnetic properties in a large area wafer can be different at different locations due to variation in film thickness, composition and growth induced residual stresses⁹⁻¹⁰. Hence, large area deposition process parameters are required to be optimized for obtaining uniform magnetic properties in Co₂FeSi thin films. This paper reports the sputtering process optimization adopted for obtaining desired magnetic properties in large area deposition of Co₂FeSi film on 3-inch size thermally oxidized Si(100)/SiO₂ wafers.

While developing sputtering process for large area thin film deposition, establishing exchange biasanisotropy in magnetic multi-layers is also important requirement for fabrication of magnetic field sensors. The exchange bias anisotropy is utilized to improve the thermal stability of the Tunneling Magnetoresistance (TMR) based magnetic field sensors. The exchange coupling at the interface of a ferromagnetic (FM) layer and an Antiferromagnetic (AFM) layer can result in the shift of the FM hysteresis loop along the magnetic field axis with respect to zero field. This phenomenon is known as the Exchange Bias (EB). This effect is characterized by an exchange anisotropy field Heb, which is often described as a result of an acting in-plane unidirectional anisotropy. The application of the exchange bias effect has gained increasingly significance; in particular, the utilisation of EB has played an important role in the development of magnetic data storage and magnetic sensor

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technologies¹¹⁻¹⁴. Ir₅₀Mn₅₀ is well known as a disordered AFM with TN of 585 K and this alloy is favored over other AFM materials in our study due to the more efficient pinning and larger exchange bias field. The realization of EB in Co₂FeSi/Ir₅₀Mn₅₀ is therefore commonly achieved by thermal processes, namely by annealing over the blocking temperature and subsequent coolingin the presence of a magnetic field. Hence, the process techniques developed for achieving magnetic exchange bias in Co₂FeSi film with antiferromagnetic Ir₅₀Mn₅₀ is also discussed in this paper.

2. EXPERIMENTAL WORK

2.1 Preparation of Sputtering Target

The preparation of sputtering target is the first step in conducting sputtering experiments. Co,FeSi alloy targets were fabricated indigenously using vacuum inducting melting followed by wire cutting using electric discharge machine (EDM). Co₅₀Fe₂₅Si₂₅ (at.%) alloy was melted using highpurity Co, Fe and Si raw materials under vacuum better than 5x10⁻⁵ mbar. The molten alloy was poured into 3-inch dia. steel mould for casting under vacuum. Then the as-cast alloy was hot pressed isostatically at 900 °C for 4 hrs in argon atmosphere to remove micro defects and to ensure chemical homogeneity. The 2 inch dia. disks were cut from the alloy using EDM wire cutting. The photographs of the as-cast alloy ingots and 2 inch dia sputtering targets are shown in Fig. 1. The composition of the target was determined using wet chemical analysis and was found to be very close to the stoichiometric composition i.e. Co_{so}Fe₂₅Si₂₅. The composition of the targetwas also analyzed with energy dispersive X-ray spectroscopy (EDS) technique. Both the analysis results confirm the correctness of the composition of the alloy target. Ir₅₀Mn₅₀ sputtering target used in this study was procured commercially.



Figure 1. Photographs of as-cast Co₂FeSi alloys prepared by vacuum induction melting and 2 inch dia., 1-5 mm thick sputtering targets wire cut from the casted alloys.

2.2 Deposition and Characterisation of Thin Films

In this study, an ultrahigh vacuum DC magnetron sputtering system was used to deposit Co_2FeSi thin films on 3-inch dia. Si(SiO₂) wafer. The sputtering parameters are Ar pressure, sputtering power, deposition time and substrate temperatures. While varying one parameter, the other parameters are kept constant. The target to substrate distance was kept constant at ~4 inch for all the deposition experiments. Prior to sputtering, the chamber is evacuated to a base pressure level of 4.5 x 10⁻⁸ Torr and backfilled with high purity (99.999 %) Argon gas to

attain the desired pressure level. Before depositing the film, the targets are pre-sputtered for 5-10 minutes to get clean fresh surface. The Co₂FeSi thin films were deposited on thermally oxidized Si(100) wafer under varying sputtering power, Ar gas pressure, sputtering time and substrate temperature in order to optimize process parameters. SiO₂ The films were deposited using 1.5 mm thick and 50.8 mm (2 inch) diameter circular Co_{so}Fe₂₅Si₂₅ (at.%) alloy sputtering target on 0.5 mm thick Si(100)/SiO, substrates. The sputtering pressure was varied, such as 2, 5, 10, 15 and 20 mTorr, with other parameters such as sputtering power, substrate to target distance and sputtering time unchanged. While the sputtering power was varied, such as 25, 50, 75, 100 and 125 W with other parameters unchanged. High purity (IOLAR-I Grade) Ar gas was used as a sputtering gas with typical flow rate of 10-12 sccm. The Co,FeSi/ IrMn multi-layers were deposited onto thermally oxidized Si substrates (Si/SiO₂) at room temperature. The sputtering parameters used are same as optimized for the large area deposition of Co₂FeSi thin films.

The thickness of the films was determined using stylus profilometer and X-ray reflectivity technique.¹⁵ Further, the stylus profilometer was also used to determine the surface roughness of the films. The crystal structure was determined by gracing incidence X-ray diffraction (GIXRD) technique. The surface morphology of the films was investigated using Atomic Force Microscope (AFM). Composition was measured using energy dispersive spectroscope (EDS) technique with those of films deposited on A12O3 substrate. From these studies, deposition conditions such as sputtering power, sputtering pressure and substrate temperature for Co, FeSi films on Si(100)/ SiO₂ substrates have been optimized. The films deposited at 5 mTorr, 50W exhibited stoichiometric Co2FeSi composition, high degree of crystalline structure and low surface roughness. Hence, further sputtering experiments were carried out on large size substrates by keeping the sputtering pressure, power and deposition time constant at 5 mTorr, 50 W and 60 min, respectively. Annealing of the films was carried out in presence of magnetic field in a vacuum heat treatment furnace. Magnetic properties of the films were studied using a vibrating sample magnetometer (VSM).



Figure 2. Variation of film thickness as a function of distance from the origin of the wafer.



Figure 3. The Co₂FeSi thin film of 50 nm thickness deposited on 3 inch size wafer and the waferwas cut into (5x5) mm squares for various measurements and the location of the samples are represented by the (x,y) coordinates.



Figure 4. AFM images of the Co₂FeSi thin film samples taken from (a) near the center, (b) near the middle and (c) near the edge of the wafer. Corresponding 2D images given below each image.

3. RESULTS AND DISCUSSION

3.1 Film Thickness Uniformity, Surface Roughness and Composition:

The Co_2FeSi thin films of 50 nm thickness were deposited on 3 inch size thermally oxidized $\text{Si}(100)/\text{SiO}_2$ wafers using the optimized sputtering conditions at room temperature. The thicknessuniformity and surface roughness of the films have been studied systematically over the 3 inch area. For thickness measurement, two diagonal lines were made on the cleaned wafer before deposition using permanent marker pen. Then after deposition, the wafer was washed in IPA to remove the marker line and to left off the film deposited on the line to form a well defined groove. The thickness of film was determined by measuring the height of the groove at different places at an interval of 5 mm from the origin. The variation of film thickness as a function of distance is shown in Fig. 2.

It can be seen that the thickness of the films was found to be uniform throughout the 3 inch wafer. The variation of thickness is ± 1 nm of 50 nm which is less than 2 %. In general, the film thicknessvaries with both the sputtering power as well as sputtering pressure. The most important parameters during sputtering are the deposition rate which depends critically on sputtering. The substrate rotation was optimized to 10 RPM for uniform deposition. The sputtering experiments were carried out at an off-axis angle of 45 deg between the target surface and substrate. Under the above conditions, the deposition rate (0.04 nm/sec) was found to be constant throughout the experiment and hence uniform film thickness is achieved over the 3 inch area.

The surface morphology of the films was studied using Atomic Force Microscopy (AFM) at different locations of the 3 inch wafer. The locations ID (samples ID) are represented by the (x,y)coordinates from the origin as shown in Fig. 3. The AFM images of the film scanned at different places are given in Fig. 4. It can be seen that all the samples are found to be continuous and exhibit uniformly distributed globular islands of fine size. No texturing effect (elongated islands)was visible in the AFM images and variations in the microstructural features are minimal.

The surface roughness was measured at two different places for each sample in both forward andreverse scans. The scan was carried out for (1x1) and (2x2) micron ranges for all samples. It was observed that the rms value of surface roughness is found to be sub-nanometer that varies from

0.42 nm to 0.75 nm throughout the 3 inch size wafer. The observed values of roughness and its variation indicate that the deposited films are very smooth over the 3 inch area. As in the case of film thickness, the sputtering power, pressure, deposition rate and substrate temperature are the important parameters which determine the surface roughness of the film. A variety of different surface morphologies and microstructures may be observed and these are inherently related to growth mechanism¹⁶. In this case the deposition was carried out at room temperature and other parameters have played an effective role. The observed values of surface roughness indicate that the films are very smooth and also the sputtering parameters used for deposition are optimised.

The chemical composition of thin film is the most important parameter determining the functional properties. Therefore, the compositional analysis is essential during development of process for large area deposition of thin films for device applications. In this study the composition of film was measured using Energy Dispersive X-ray Spectroscopy (EDS) attached to FESEM. The compositional details of the Co_2FeSi film measured at different places of 3 inch size wafer are given in Table 1. It can be observed that the composition of film is uniform throughout the wafer. At the center and middle of the wafer the composition of Co, Fe and Si are very close to the stoichiometric composition and their variation is within the experimental accuracy. However, there is a little variation

Table 1.Composition of Co_2FeSi films deposited on 3 inch
size Si (100)/SiO2 wafer measured at different places
using EDS technique (Q1,2,3,4 are four quadrants of
the wafer).

Sample ID	Sample location	EDS film composition (at.%)			_ Remark
	F	Co	Fe	Si	
(1,1)	Wafer Centre (Q1)	51.0	24.8	24.2	
(-1,-1)	Wafer Centre (Q3)	50.6	24.9	24.5	
(-3,4)	Wafer Middle (Q2)	51.4	24.5	24.1	
(3,-3)	Wafer Middle (Q4)	50.5	25.0	24.5	
(1,7)	Wafer Edge (Q1)	49.5	25.2	25.3	PW : 50 W
(-1,6)	Wafer Edge (Q2)	49.2	25.1	25.7	$P_{Ar}=5 \text{ mTorr}$ Th= 50 nm
(-1,-7)	Wafer Edge (Q3)	49.9	24.0	26.1	1 II— JU IIIII
(1,-7)	Wafer Edge (Q4)	48.2	24.1	27.7	

in the element composition at the edges of the wafer. The concentration of Co, Fe is slightly depleted while the Si content is enriched at the edges. This may be due to the differentsize of atoms. The Co and Fe atoms are heavier than Si and they get scattered strongly by residualArgon gas molecules compared to the Si atom and hence more Si atoms were arrived up to the edges of substrate result in a higher Si concentration. However, this region of wafer is normally excluded during the micro fabrication of devices using large size wafers. Hence, these results indicate that the composition of film is uniform in the wafer for the extent which is used for the device fabrication in 3 inch size wafer.

3.2 Magnetic Properties

The magnetic anisotropy (K_n), coercivity (H_n), squareness ratio (S), remanent magnetization (M) and saturation magnetization (Mss) are the important magnetic properties of Co,FeSi thin films. Magnetic properties of the films were analyzed using Vibrating sample magnetometer (VSM) by applying magnetic field parallel to the plane of film at room temperature. Angular dependence of the magnetic properties was also measured by varying angle of in-plane magnetic field with respect to (100) direction to study the magnetic anisotropy. The typical M-H hysteresis loops of Co, FeSi thin films diced from 3 inch wafer at different locations are shown in Fig. 5. Figure 5 clearly indicate that all films, irrespective of the locations, display ferromagnetic hysteresis loopsand are well saturated below ~ 100 Oe. The coercivity values were found to be less than 100 Oe and these values vary only from 85 to 92 Oe for the films diced from centre of the 3 inch wafer. Whereas the coercivity values are varied from 21 to 99 Oe for the films diced from the middle and edges of the wafer. A similar variation was also observed in saturation and remanent magnetizations. A maximum saturation magnetization, Ms value of 990 emu/ cc was observed in the sputtered Co₂FeSi thin film which is slightly less than that of 1040 emu/cc of the bulk alloy. This difference in magnetization may be attributed to the surface effect of thin film and the magnetic interaction of the atoms



Figure 5. M-H hysteresis loops of Co₂FeSi thin films deposited on 3 inch thermally oxidized Si (100) wafer. The measurements were carried out at different locations by dicing the wafer (5x5) mm size square samples.

Table 2.	Magnetic properties such as coercivity (H _c), saturation magnetization (M _s), remanent magnetization (M _r), squareness
	ratio (Sr) of Co ₂ FeSi thin film deposited on 3 inch Si(100)/SiO ₂ wafer measured at different locations by dicing (5x5) mm
	square samples from the wafer.

Sample ID	θ (deg)	H _c (Oe)	M _s (emu/cc)	M _r (emu/cc)	S _r Ratio
(1,1)	0	91.0	641	416	0.65
	90	92.1	609	407	0.66
(1,-1)	0	89.6	688	644	0.93
	90	90.0	665	625	0.94
(-1,1)	0	85.7	662	567	0.85
	90	87.7	656	627	0.94
(-1,-1)	0	88.0	597	559	0.94
	90	90.3	590	552	0.93
(4,-1)	0	91.4	822	783	0.95
	90	74.8	736	605	0.82
(1,-3)	0	89.2	622	569	0.91
	90	86.4	562	501	0.89
(1,-5)	0	65.6	990	662	0.67
	90	98.9	873	858	0.98
(-4,-1)	0	96.1	607	540	0.89
	90	77.9	608	434	0.71
(-3,3)	0	21.2	735	322	0.44
	90	24.5	661	376	0.57
(())	0	39.1	884	877	0.99
(-0,-2)	90	24.2	867	062	0.07

near the surface is expected to be weaker due to the reduced atomic coordination and hence the lower magnetisation.

The details of magnetic properties obtained from different locations of 3-inch wafer are also given Table 2. These values are in good agreement with the values reported in literature¹⁰.

The room temperature magnetization studies revealed that all the films are found to exhibit ferromagnetic properties with low coercivity. However, the magnetic properties are not uniform throughout 3 inch wafer and the values of coercivity, saturation magnetization and shape of M-H hysteresis loops are found to be different at different locations though the film thickness, surface roughness and composition were uniform throughout the 3-inch wafer. It was observed that Co₂FeSi thin film deposited on Si(100)/SiO₂ wafer exhibits nearly isotropic magnetic properties near the centre but in-plane magnetic anisotropy was observed at middle and edges of the wafer. The angular dependence of magnetization curves also revealed that the magnetic properties are anisotropic in nature, which is not desirable for device fabrication. The magnetic anisotropy may arise from the crystal structure of the film or growth induced residual stresses during the deposition of film due to the lattice mismatch and difference in coefficient of thermal expansion between the film and substrate. The coercivity of magnetic thin films is influenced by different parameters suchas magnetocrystalline anisotropy, shape anisotropy, strain due to residual stress, film roughness, grain size etc. When the film is

subjected to any stress, its domain dynamics also gets effected which results in change in coercivity. The crystal structure of Co_2FeSi is cubic and crystalline magnetic anisotropy is small and hence the residual stresses are expected to play a key role in thefilm. This was confirmed by depositing the film on (10x10) mm size smaller wafers placed at different locations within 3 inch area as shown in Fig. 6. These films showed isotropic magnetic properties as the stresses were relaxed in the smaller size wafer and hence uniform magnetic properties were obtained.

In order to overcome this anisotropic magnetic behavior, a Cr buffer layer was deposited on the Si(100)/SiO₂ wafer before the deposition of Co₂FeSi film. The Cr buffer layer was found to be most suitable for growing contact layer for CFS films also. The Cr films of different thicknesseswere deposited on thermally oxidized Si(100) wafer and the surface roughness was studied using both stylus profilometer and atomic force microscopy. The rms roughness value of the film is found to be minimum (~0.64 nm) for the film having thickness of 15 nm and hence Cr buffer layerof 15 nm is used for depositing Co₂FeSi films. The CFS films deposited on Si(100)/SiO₂ wafer with Cr buffer exhibited nearly uniform magnetic properties throughout the 3 inch wafer.

The typical M–H hysteresis loops of Co_2FeSi thin film deposited on 3 inch size $Si(100)/SiO_2$ waferwith 15 nm thick Cr buffer layer are shown in Fig. 7. It can be seen that all the



Figure 6. M-H hysteresis loops of Co₂FeSi thin films deposited on 3 inch thermally oxidized Si (100) wafer. The measurements were carried out at different locations by dicing the wafer (5x5)mm size square samples.



Figure 7. Angle dependent M–H hysteresis loops of Co₂FeSi thin films with 15 nm Cr buffer layer (a) taken at (1,1) and (b) taken at (1,-7) location. Only 0 and 90 deg M-H loops are shown as otherdeg loops are similar to these angles.



Figure 8. Polar plots representing angle dependent coercivity of Co₂FeSi thin films deposited on 3 inch size (a) Si(100)/SiO₂ wafer and (b) Si(100)/SiO₂ wafer with 15 nm Cr buffer layer.

M-H hysteresis loops of films diced at different locations are similar and indicate magnetic properties are isotropicin nature. The coercivity values measured at different locations vary from 100 to 120 Oe and thissmall variation indicates that the magnetic softness of film is uniform. However, the observed coercivity values were found to be slightly higher than that of the thin film without Cr buffer layer. A higher coercivity may be attributed to the anti-ferromagnetic coupling between the Cr atom and the film at the interface. An interface roughness may also increase the coercivity of film as it restricts the free movement of magnetic domains and hence a slight increase in the coercivity is observed. The saturation magnetization values vary from 525 to 600 emu/cc measured at different locations. These values are much less than that of the bulk Co₂FeSi alloy (1040 emu/cc) and the thin film (990 emu/cc) without Cr buffer layer. A lower value magnetization is not fully understood, however, it may be related to the effect of Cr buffer layer and diffusion of Cr atom in the film. The diffused Cr atoms will couple anti-ferro magnetically with Co and Fe atoms in the film and it may lead to decrease in magnetization. However,

the variation in magnetizations measured at different locations is small and it indicates that the magnetic properties of the film areuniform throughout the 3 inch wafer.

To understand the magnetization reversal and the role of magnetic anisotropy, the angulardependence of coercivity of the film with and without Cr buffer layer was studied. The magnetic anisotropy decides the preferred crystallographic direction of magnetization and normally, in the thin films, the nature of magneto-crystalline anisotropy is dictated by the underlying crystallographic symmetry of the material. The cubic Co₂FeSi alloy thin films exhibit a dominant in-plane uniaxial anisotropy and it strongly depends on the process parameters of the film growth. The M-H hysteresis loops measured at different inplane magnetic field angles and correspondingpolar coercivity plots for the Co₂FeSi films with and without Cr buffer layer are shown in Fig. 8. In the film without Cr buffer, rectangular hysteresis loops are observed when the external magnetic field is applied along the easy axis of magnetization [(100)-axis] and these loops are characterized by the normalized squareness ratio close to 1, the squareness ratio is defined as M_r/M_s , where

 M_r is the remanent magnetization and M_s is the saturation magnetization. These loops also exhibit a maximum coercive field (H_c) and H_c go through two maxima at 0 and 180 degrees. Thus, the two-fold anisotropic magnetic properties were observed in the film without Crbuffer which is not desirable for the device applications.

On the contrary, the Co₂FeSi film with Cr buffer layer does not exhibit any peak in the polar plot of coercivity. The coercivity does not change with the angle of magnetic field applied during measurements. A circular variation in coercivity was observed in the polar plot of the filmswith Cr buffer and indicated that the coercivity of the film is uniform throughout the 3 inch size wafer. When the films were deposited with buffer layer, the effective residual stress level on the films could be reduced and make nearly uniform. This resulted in obtaining uniform magnetic properties throughout the 3-inch dia. size wafer. Hence, the polar plot of coercivity does not exhibit any peak. The isotropic nature of magnetic properties such as coercivity and saturation magnetization of the film with Cr buffer is desirable for device applications¹⁷⁻¹⁹.

3.3 Exchange Bias Anisotropy Studies on Co₂FeSi/ IrMn Multilayers

The exchange bias interaction is established in ferromagnetic and anti-ferromagnetic multi-layersby magnetic annealing the multi-layer stack above the Neel temperature (T_{y}) of anti-ferromagnetic layer. The magnetic annealing is carried out at a constant temperature in presence of magnetic fieldin a vacuum furnace followed by cooling the samples in the field. The annealing temperature, time and strength of magnetic field are the critical parameters which controls the strength of exchangebias interaction in the multi-layers. The in-house made magnetic annealing set-up used in the present study is shown in Fig. 9. The Sm₂Co₁₇ permanent magnet and Fe soft magnetic yoke were used in the set up as source of magnetic flux and flux concentrator respectively. A maximum magnetic field obtained in the air gap is 1000 Oe. The sample is fixed on the Al holder kept in theair gap between the permanent magnets and loaded in to the vacuum furnace for annealing.



Figure 9. In house made magnetic field annealing set-up used in vacuum furnace.

Magnetic properties of EB was measured by vibrating sample magnetometer (VSM) in the present study, exchange bias field H_{EB} or $H_{ex} = (H_{C1}+H_{C2})/2$ and coercivity $H_{C} = (-H_{C1}+H_{C2})/2$, where H_{C1} and H_{C2} denote the left and right



Figure 10. AFM images of Ta(10nm)/Cu(15nm) buffer layer showing smooth surface withroughness less than 0.5 nm.

coercivity from the M-H hysteresis loop²⁰⁻²¹.

The Si/SiO₂ /Ta(10 nm) /Cu(15 nm) /IrMn(10 nm)/ Co₂FeSi(10 nm) /Ta(5 nm) multi-layers deposited by UHV sputtering were studied. Ta(10 nm)/Cu(15 nm) was deposited as buffer layer on Si/SiO₂ substrate to have smooth surface and then Ta(5 nm) was deposited for capping layer to protect thestack from oxidation. The low surface roughness (less than 0.5 nm) is an important requirement toachieve high exchange bias field. The Ta/Cu buffer layer is known to provide smooth surface on Si/SiO₂ substrate and surface roughness of each layer was measured using AFM before deposition of next layer. The typical AFM images of Ta/Cu buffer layer are shown in Fig. 10. It can be seen that the surface is very smooth with roughness is found to be less than 0.5 nm.

The multi-layer stacks were annealed at different temperatures, say 150, 200, 250 and 300 °C in presence of magnetic field of 1000 Oe using vacuum furnace for 1 hr and subsequent cooling to room temperature in presence of magnetic field. The M-H hysteresis loops of annealedsamples were measured using VSM at room temperature and are shown in Fig. 11.

The magnetic field was applied parallel to the film plane during annealing as well as hysteresis loop measurements. The exchange bias parameters such as exchange bias field (H_{ex}) and coercivity (H_c) obtained from the M-H loops are also represented in a plot as given in Fig. 11(f). It is clearly seen that the as-deposited film does not show any shift in the M-H loop whereas the annealed filmsare shifted in the field axis (x-axis) from zero field. It clearly shows that the films annealed in magnetic field exhibit exchange bias effect.

When the sample is cooled in presence of magnetic field, the anti-ferromagnetic spins gets aligned along with the ferromagnetic layer and it acts as a biasing layer. The exchange field exerted by the anti-ferromagnetic layer at the interface is biasing the ferromagnetic layer and hence the M-H hysteresis loop of ferromagnetic layer is shifted along the field axis. The shift is proportional to the strength of exchange bias field exerted by anti- ferromagnetic layer at the interface. The exchange field is increased as the annealing temperature is increased up to 200 °C and then it starts to decrease upon further increase in annealing temperature. The increase in H_{ex} may be attributed to the increase in inter layer coupling and improved crystalline nature of the layers. The Hex field is known to be an oscillatory field and hence it decreases at higher annealing temperature



Figure 11. M-H hysteresis loops of (a) as deposited Co₂FeSi/IrMn multilayers, (b) to (d) magneticannealed at various temperatures. (f) Variation of exchange bias field and coercivity as a function of annealing temperature is shown at right bottom corner.

even though magnetic field and annealing time are the same. The exchange bias field is also considered as the unidirectional anisotropy and hencethe presence of exchange anisotropy field is expected to increase the coercivity of the sample.

It can be seen from Fig. 11(f) that the coercivity, H_c was found to increase from 90 to 135 Oe as the annealing temperature increases to 300 °C except for the 200 °C. A maximum Hex field of 178 Oe and minimum coercivity of 85 Oe were achieved for the 200 °C annealing temperature and 1000 Oe magnetic field, which are optimum conditions for establishing exchange bias anisotropy in the Co₂FeSi/IrMn bilayers. This is because in this system, anti ferromagnetic coupling with ferromagnetic layers increases up to 200 °C (close to Neel temperature of the antioferromagnetic film). This results in increase in Hex with temperature up to 200 °C. Further increase in temperature results in increasing interfacial roughness, thereby decreasing the H_{ex} .

4. CONCLUSIONS

• Indigenously fabricated Co2FeSi alloy and commercially

procured IrMn alloy sputtering targets of 2 inch diameter circular disks were utilized for sputtering experiments in the present work. The sputtering process parameters for large area deposition of Co₂FeSi magnetic thin films on 3 inch size thermally oxidized single crystal Si(100)/SiO₂ wafers have been developed by optimizing the sputtering geometry and other process parameters

- The DC power, Ar gas pressure, gas flow rate, substrateto-target distance, substrate rotation and sputtering time are other parameters optimized in the process flow. The optimized sputtering parameters viz. 50 W and 5 mTorr sputtering power and pressure respectively were used for deposition of Co₂FeSi films
- A uniform film thickness, composition, smooth surface, good crystallinity and magnetic properties have been achieved in the films over 3-inch size wafers with less than 2% variations in film thickness. The isotropic magnetic properties such as saturation/remanent magnetizations, coercivity are achieved for Co₂FeSi films deposited on 3 inch size Si(100)/SiO₂ wafers with Cr buffer layer. This could possibly be due to the reduction in residual stress level on thin films due to buffer layer
- An exchange bias anisotropy has been established in Co₂FeSi/IrMn multilayers by magnetic annealing process using in-house made magnetic annealing set up. A maximum exchange bias field, Hex of 178 Oe and low coercivity, Hc of 85 Oe has been achieved in the above multilayer stacks suitable for magnetic tunnel junctions
- The developed sputtering process flow, device quality large area thin films and exchange bias multilayer stacks were found to be useful for the fabrication of magnetic tunnel junctions (MTJ) on 3 inch size Si(100)/SiO₂ wafer for magnetic field sensor applications.

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