Magnetic Structure Characterization Over Multiple Length Scales

Magnetization Reversal and Domain Structures in Perpendicular Synthetic Antiferromagnets Prepared on Rigid and Flexible Substrates

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Ferromagnetic (FM) layers separated by nonmagnetic metallic spacer layers can exhibit Ruderman-Kittel-Kasuya-Yosida (RKKY) coupling, which may lead to a stable synthetic antiferromagnetic (SAF) phase. In this article, we have studied magnetization reversal by varying the number of bilayer stacks \([\text{Pt}/\text{Co}]\) as well as thicknesses of Ir space layer \(t_{\text{Ir}}\) on rigid Si(100) and flexible polyimide substrates. The samples with \(t_{\text{Ir}} = 1.0\) nm show a FM coupling, whereas samples with \(t_{\text{Ir}} = 1.5\) nm show an antiferromagnetic (AFM) coupling between the FM layers. At \(t_{\text{Ir}} = 2.0\) nm, it shows a bow-tie shaped hysteresis loop indicating a canting of magnetization at the reversal. Higher anisotropy energy compared to the interlayer exchange coupling (IEC) energy is an indication of the smaller relative angle between the magnetization of lower and upper FM layers. We have also demonstrated the strain-induced modification of IEC as well as magnetization reversal phenomena. The IEC shows a slight decrease upon application of compressive strain and increase upon application of tensile strain, which indicates the potential of SAFs in flexible spintronics.

INTRODUCTION

Tunability, miniaturization and functionality of spintronic devices depend on several factors including the interface engineering of ferromagnetic (FM) and nonmagnetic (NM) ultrathin layers. Recently, the synthetic antiferromagnet (SAF) has drawn immense research interest because of its advantage over antiferromagnetic (AFM) materials.\(^1\) It consists of two FM layers separated by a metallic spacer layer, having antiparallel magnetization among the consecutive FM layers.\(^1,2\) The FM layers are coupled by interlayer exchange coupling (IEC), which is the RKKY type exchange coupling. This coupling is of oscillatory nature and decays with the NM layer thickness. Therefore, in a FM/NM/FM system, global FM or AFM behavior is seen depending on the thickness of the spacer.\(^3–6\)

SAFs can be used as a key component of magnetic tunnel junction (MTJ) devices for better data retention and thermal stability due to the absence of a stray field. In MTJs the stray field arising from the reference layer affects the free layer, hindering parallel or antiparallel alignment of the magnetization. Using a SAF with reduced stray field, the reference layer becomes pinned, which may lead to a stable MTJ.\(^2,6,7\) In SAFs, e.g., in Co/Ni layers with Ru spacer, higher domain wall velocity has been observed.\(^8\) Furthermore, SAFs are becoming promising nowadays as they host chiral magnetic...
textures viz. skyrmions. It has been shown that IEC between two FM layers helps to minimize the skyrmion Hall effect. One of the important aspects of the SAFs is that the properties can be manipulated by applying strain to the sample. Vemulkar et al. have shown the modification of RKKY coupling in a perpendicular SAF prepared on a flexible substrate, which also shows a robust switching behavior. Yang et al. have demonstrated the voltage-controlled domain evolution in SAFs on flexible substrate. Flexible electronics have shown major technological innovation recently. The fabrication of electronic devices on flexible substrates has led to the development of a bendable electronic skin and displays. Preparation of the SAF with perpendicular magnetic anisotropy (PMA) on flexible substrates requires special attention since the quality of the interface between layers strongly depends upon the roughness of the substrate and thickness of the buffer layer. Hence, preparing such thin films on flexible substrates such as polyimide is an important step to proceed further in this direction.

In this context, we have fabricated SAFs by taking Co as FM layer and Ir as spacer layer deposited on both rigid and flexible substrates. For the SAFs with Ir thickness of 1.0, 1.5 and 2.0 nm fabricated on rigid Si(100) substrates, we have observed three different types of spin configuration among the FM layers, i.e., FM coupling, AFM coupling and canted magnetic configuration, respectively. The effect of such inter-layer couplings on magnetization reversal and domain structure has been investigated. Furthermore, we have performed a similar study for a SAF sample deposited on a flexible polyimide substrate.

**EXPERIMENTAL DETAILS**

Nine samples have been prepared on rigid Si(100) substrates with sample structure Si/Ta(3)/[Pt(3.5)/Co(0.8)]m/Ir(1.5)/Co(0.9)/Ir(1.5)/Co(0.9)/Ta(3). All the thicknesses shown in the parentheses are in nm. In the sample structure, numbers of stacks of [Pt/Co] layers below and above the Ir spacer, i.e., (m, n), are (2, 1) and (1, 1), respectively, for tIr = 1.0, 1.5, and 2.0 nm. We name these six samples as S-2-1-1.0-1, S-2-1-1.5-1, S-2-2-2.0-1, S-1-1-1.0-1, S-1-1-1.5-1 and S-1-2-2.0-1. Another two samples with tIr = 1.5 nm and (m, n) as (1, 2) and (2, 2) are named S-1-1-1.5-2 and S-2-1-1.5-2, respectively. The sample names and their structures are listed in Table I. A schematic of the sample structure is shown in Fig. 2a. A seed layer of Ta is grown on the substrate. It favors the growth of Pt layer to provide PMA in the Co layer. In all the samples (except reference one), Pt is used as a capping layer to prevent oxidation of the Co layer.

To study the effect of stress on the strength of IEC for the magnetic multilayers, another sample has been prepared on 38 μm thick flexible polyimide (PI) substrate (manufactured by Dupont). The sample structure is Pt/Ta(15)/Pt(3.5)/Co(0.9)/Ir(1.5)/Co(0.9)/Pt(3.5). All the thicknesses shown in the parentheses are in nm. This sample is named F-1-1.5-1. Here, the Ta seed layer is taken as 15 nm to reduce the effect of roughness of the polyimide substrate on the grown film. All the samples have been prepared in a high-vacuum multi-deposition chamber manufactured by Mantis Deposition Ltd., UK. The base pressure of the chamber was > 1 × 10⁻⁷ mbar. The deposition pressure was ~ 1.5 × 10⁻³ mbar for Ta and Pt layers. Furthermore, for Co and Ir, the deposition pressures were ~ 5 × 10⁻³ mbar and ~ 2.1 × 10⁻³ mbar, respectively. During sample preparation, the substrate table was rotated at 15 rpm to minimize the growth-induced anisotropy and also to have uniform growth of the films. The rates of deposition were 0.1 Å/s, 0.13 Å/s, 0.3 Å/s and 0.1 Å/s for Ir, Ta, Pt and Co, respectively.

For the structural characterization of these ultrathin layers, we have performed cross-sectional TEM imaging in a high-resolution transmission electron microscope (HRTEM) (JEOL F200, operating at 200 kV and equipped with a GATAN onewview CMOS camera) in the STEM mode. For the cross-sectional TEM sample preparation, two small pieces of samples were attached with film sides facing each other by using epoxy glue. After several processes of cutting, grinding and dimpling using a diamond wire cutter, disc grinder and dimple grinder, the processed 3-mm-diameter sample was milled with Ar ion in the PIPS (Precision Ion Polishing System) II manufactured by GATAN. The milling was performed with an ion beam energy of 5 keV and a milling angle of 4° both below and above by two ion guns in the dual modulation mode.

Simultaneous observations of magnetic domain images and hysteresis loop in polar mode have been performed for all the samples with the help of a magneto-optic Kerr effect (MOKE)-based microscope manufactured by Evico magnetics GmbH, Germany. For quantifying the IEC energy and anisotropy energy, etc., we have performed the hysteresis measurements at room temperature using a SQUID-VSM (Superconducting Quantum Interference Device-Vibrating sample magnetometer) manufactured by Quantum Design, USA.

To generate tensile and compressive strains, the flexible sample has been fixed using Kapton tape on both convex- and concave-shaped molds, respectively. The magnitude of strain is varied by using molds of different radii of curvatures. To apply tensile strain on the sample, the flexible film is attached on the convex mold with the film being upside as shown in Fig. 1a. The top portion of the film experiences a tensile strain whereas the bottom portion experiences a compressive strain. A
schematic of this phenomenon is shown in the Fig. 1d. A neutral layer exists at the middle of the total thickness where the system experiences no strain. Therefore, a tensile strain is experienced by the Co/Pt film whereas the PI substrate experiences a compressive strain when mounted on a convex mold.

Furthermore, to apply a compressive strain on the flexible film, it is attached on the concave mold as shown in Fig. 1c. Here, the Co/Pt film on the upside experiences a compressive strain whereas the PI substrate faces a tensile strain.

**RESULT AND DISCUSSION**

Figure 2b shows the high-resolution STEM images of sample S-2-1.5-1. The growth of individual layers is clearly visible (indicated in the image with respective layer names). The corresponding EDS line scan is shown in Fig. S1 of the supplementary information.

Hysteresis loop of the reference sample R1 measured by SQUID-VSM in presence of a perpendicular magnetic field is shown in the Fig. S2 of supplementary information. Magnetic hysteresis loops of samples S-2-1.5-1 and S-1-1.5-1 are shown in Fig. 2c and 2d, respectively.
in Fig. 3a and b, respectively, measured by SQUID-VSM. The steps in the hysteresis loops indicate AFM coupling between the FM layers below and above the Ir spacer layer. Figure 3a shows that the magnetization reversal is accompanied by three steps in the hysteresis loop. Here, the two Co layers below the Ir spacer layer behave such that they are coupled ferromagnetically and reverse simultaneously during the magnetic field sweeping. However, the Co layer above the Ir layer reverses separately. The magnetization reversal may be explained in the following manner. In the first reversal the top FM layer switches first and becomes AFM coupled to the bottom FM layers. In the second reversal, both the top and bottom FM units switch oppositely because of strong AFM coupling. Finally, under sufficiently negative external field (Zeeman energy), the top layer switches along the negative field direction, and hence saturation is achieved. For sample S-1-1.5-1, there are only two reversals, indicating that in the first reversal, both layers become AFM coupled, and in the second reversal, the coupling is lost and both FM layers become negatively saturated. Furthermore, we observe in Fig. 3a that there is a substantial remanent magnetization in the sample indicating that S-2-1.5-1 is an uncompensated SAF, but in Fig. 3b, the remanence is almost close to zero, which indicates that S-1-1.5-1 is a compensated SAF. The other two samples, S-1-1.5-2 and S-2-1.5-2, also show the AFM coupling between the FM layers, which is shown in the supplementary information. The IEC energy of these AFM coupled FM layers can be calculated by using the expression $J_{ex} = H_{ex}M_s t$ where $H_{ex}$ is the exchange coupling field at which the coupling between the FM layer
vanishes and the magnetization directions in both the layers become parallel.\textsuperscript{21,22} $M_s$ is the saturation magnetization and $t$ is the thickness of the FM layers.\textsuperscript{23} $H_{ex}$ can be calculated from the hysteresis loop measured by SQUID-VSM as indicated in Fig. 3a. The IEC energy ($J_{ex}$) of AFM coupled samples S-2-1.5-1, S-1-1.5-1, S-1-1.5-2 and S-2-1.5-2 is found to be $2.96 \times 10^{-4}$, $1.64 \times 10^{-4}$, $2.74 \times 10^{-4}$ and $4.56 \times 10^{-4}/m^2$, respectively. Here, it shows an increase in the coupling strength with the increase in the number of Co/Pt layers indicating that it needs more energy to break the coupling for more Co/Pt layers below and above the spacer. Samples S-2-1.0-1 and S-1-1.0-1, with $t_{Ir} = 1.0$ nm, show FM coupling indicating that this thickness of Ir is not in the AFM coupling regime. M-H loops corresponding to these samples are shown in supplementary information. The other two samples, S-2-2.0-1 and S-1-2.0-1, with $t_{Ir} = 2.0$ nm, show a bow-tie shape hysteresis loop with no steps. This indicates that the AFM coupling is reduced at this Ir thickness.

The effective anisotropy energy values for all the samples have been evaluated by measuring the hysteresis loops along both in-plane and out-of-plane directions of the samples in the SQUID magnetometer. In this context, we have used the Eq. $K_{eff} = \mu_0 H_k M_s/2$, where $\mu_0 H_k$ is the anisotropy field (in-plane saturation field) and $M_s$ is the saturation magnetization (refer to Table S2 in supplementary information). We found an enhancement in the anisotropy energy of the samples with AFM coupling compared to the samples with FM coupling. The anisotropy energies of these samples are shown in Fig. 4 where the points inside the ellipse show the effective anisotropy energy values for the SAF samples. The anisotropy energy of the samples are higher than the IEC energy ($J_{ex}/t$) resulting in a smaller intermediate angle between the magnetic moments of the two layers.\textsuperscript{20,24}

Figure 5 shows the hysteresis loops and the corresponding domain images measured by MOKE microscopy in polar mode for the AFM coupled samples S-2-1.5-1, S-1-1.5-1, S-1-1.5-2 and S-2-1.5-2. From left to right, the hysteresis loops represent the samples S-2-1.5-1, S-1-1.5-1, S-1-1.5-2 and S-2-1.5-2, respectively, and below each loop the domain images are shown and the respective field points are mentioned in the hysteresis loop.

For sample S-2-1.5-1, the hysteresis has three steps as shown in Fig. 5a. Here, the magnetization reversal is achieved by both domain nucleation and domain wall (DW) propagation (Fig. 5a1–a4). Here, we have defined the magnetization directions for the Pt/Co layers above and below the Ir spacer layer by a red arrow ($\uparrow$) and blue arrow ($\uparrow$), respectively. In the first reversal ($\uparrow \downarrow - \downarrow \uparrow$), no domain has been observed and only a change in the contrast of domain image is there. This type of domain behavior may be explained as the spin flop transition, which can be inferred from the slanted reversal of the hysteresis loop at the first reversal.\textsuperscript{25,26} However, in the second reversal ($\downarrow \uparrow - \uparrow \downarrow$), bubble domains have been observed because of the sharp transition (Fig. 5a2). In the third reversal ($\downarrow \downarrow$), the size of the domains becomes remarkably small, which may be due to the lowering of AFM coupling between the layers (Fig. 5a3).\textsuperscript{27} In sample S-1-1.5-1, only two-step reversal (see Fig. 5b) has been achieved via a large nucleation of small bubble domains (Fig. 5b2–b3).

In sample S-1-1.5-2, a three-step hysteresis loop is observed, which is shown in Fig. 5c. Here, two different types of domains are observed at two different reversals, i.e., distorted bubble domain with a higher number of nucleations and symmetric bubble domain as shown in (c2) and (c3), respectively. Here, the bubble domains are quite similar to sample S-2-1.5-1 because of their comparable IEC strength. Sample S-2-1.5-2 exhibits a multi-step magnetization reversal where the reversal process is accompanied via first no domains and afterwards bigger domains. Finally, the reversal is completed via smaller bubble domains with more nucleations.

Samples S-2-2.0-1 and S-1-2.0-1 show a bow-tie shaped hysteresis loop measured by magneto-optic Kerr effect microscopy shown in Fig. 6a and c. At the reversal, very small ripple kinds of domains are observed, indicated in Fig. 6b–d and f–h. The hysteresis loops for sample S-2-2.0-1 and S-1-2.0-1 shown in Fig. 6 have been measured using a 5× objective. However, due to very small domain size, the domain images are captured using a 50× objective (as shown in Fig. 6).

To understand the effect of strain on the magnetization reversal, henceforth we will discuss the results on sample prepared on flexible PI substrate. Figure 7 shows the Kerr microscopy hysteresis loops of SAF prepared on polyimide substrates at both strained and unstrained states. In addition, the hysteresis loop of its rigid counterpart (sample

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**Fig. 4.** Effective anisotropy energy density calculated for the samples prepared on rigid Si substrates.
deposited on Si substrate) is also measured (see supplementary information) where sharp switching fields are observed for samples prepared on both the substrates.

The sample prepared on the PI substrate have always been measured upon a mold (flat, concave or convex of 4 mm height) made of aluminum, whereas its rigid counterpart was measured without using any mold. Hence, the difference in coercivity observed for the samples on Si and PI originates because of the height of the mold. We found that when the mold is used, this means the sample height is lifted with respect to the center of the magnetic coil in the Kerr microscope and more field is needed to saturate the sample. This may lead to an enhancement of coercivity. This means the SAF samples with similar structure deposited on rigid Si or flexible polymide have similar coercivities as the unstrained case (for more details, see Fig. S7 in the supplementary information).

Comparable coercive fields for the same sample on both the substrates indicate that the strength of the magnetic anisotropy and RKKY coupling is identical, which makes flexible SAFs very promising from the application viewpoint. The strain is applied to the flexible sample in the manner described in the
The magnitude of strain generated in this manner can be calculated by the following Eq. 19, 28–30
\[
\epsilon_{\pm} = \frac{t_{\text{total}}}{2R + t_{\text{total}}} 
\]
where \( \epsilon_+ \) denotes the applied tensile strain and \( \epsilon_- \) is the compressive strain, \( R \) is the radius of the mold used, and \( t_{\text{total}} \) is the total thickness of the magnetic thin film and flexible substrate.

Figure 7a shows the hysteresis loops of sample F1-1-1.5-1 under both flat and bend states. It shows a slight change in coercivity under both compressive and tensile stresses. However, the effect of strain on modifying the first and second magnetization reversal is the opposite to that shown in the inset of Fig. 7a. Under 0.37\% tensile stress, we observe an increase in coercivity of 2.03 mT while the compressive stress decreases the coercivity by 4.41 mT. Hence, only a slight modification (\(\approx 4\%\)) in coercivity has been observed for the sample under a moderate strain (\(\approx 0.4\%\)). The change in coercivity is attributed to the change in PMA as a result of the introduction of stress-induced anisotropy. 31 The stress-induced anisotropy is given by
\[
E_{\sigma} = \frac{3}{2} \lambda_s \sigma \sin^2(\theta) 
\]
where \( \lambda_s \) is the magnetostriction coefficient, \( \sigma \) is the applied stress, and \( \theta \) is the angle between the \( \sigma \) and magnetization vector. Earlier reports showed that the \( \lambda \) is negative for Co. 32
For a negative magnetostrictive FM material, the stress-induced anisotropy acts along the in-plane direction for compressive strain and out-of-plane direction for tensile strain. As a result, the stress-induced anisotropy reduces the PMA of SAF under compressive stress and enhances the anisotropy in the case of tensile stress. Figure 7b shows the variation in coercive field by varying the magnitude of both types of strain. With the increase in compressive strain, there is a systematic decrease in coercivity while with tensile strain the coercivity increases continuously. Such changes in coercivity under strain can be attributed to a change in PMA because of magneto-elastic anisotropy. The interlayer exchange coupling field ($\mu_0 H_{ex}$) has also changed under the effect of strain, which is a clear indication of the change in coupling energy. The IEC field for flat state is 73.95 mT. With the effect of tensile strain, the $\mu_0 H_{ex}$ increases to 75.62 mT and decreases to 70.91 mT in case of compressive strain. However, no significant changes of domain images have been observed under application of both types of strain shown in Fig. 8.

CONCLUSION

In this work, SAF samples with PMA have been prepared on both rigid and flexible substrates. The variation of iridium spacer layer thickness leads to different types of coupling among the FM layers with different spin configurations, i.e., FM and AFM couplings. Comparison of IEC energy and anisotropy energy suggests the relatively smaller angle between the spins of FM layers below and above the spacer layer in SAF samples. On the other hand, the effect of strain has shown a substantial change of the exchange coupling field of the SAF sample, which leads to the change of interlayer exchange coupling energy. The exchange coupling field shows an increase upon application of tensile strain and decrease upon application of compressive strain. We further noticed that the application of strain does not lead to any significant change of domain structure in the SAF sample. Future work is required to understand the detailed reversal processes in such SAFs. The effect of stress on the PMA and AFM exchange coupling between the FM layers is also studied, which may be helpful for future flexible spintronic devices.

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CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.
SUPPLEMENTARY INFORMATION

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