Giant Spin Hall angle in Iridium Oxide

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Large charge-to-spin conversion (spin Hall angle) and spin Hall conductivity are prerequisite for development of next generation power efficient spintronic devices. In this context heavy metals (e.g. Pt, W etc.), topological insulators, antiferromagnets are usually considered because they exhibit high spin-orbit coupling (SOC). However it remains a challenge to find suitable materials which exhibit high spin Hall angle and spin Hall conductivity. In this regard 5d transition metal oxide e.g. is a potential candidate which exhibits high spin Hall resistivity. Here we report a study of spin pumping and inverse spin Hall effect (ISHE), via Ferromagnetic Resonance (FMR), in Iridium Oxide (IrO$_2$)/CoFeB bilayers. We identify the individual contribution of spin pumping and other spin rectification effects in the magnetic layer, by investigating the in-plane angle dependence of ISHE signal with the external magnetic field. Our analysis shows significant contribution of spin pumping effect to the ISHE signal. We have evaluated the spin Hall angle to be $\sim 3.7$.

I. INTRODUCTION

With charge based electronics reaching their limitations, more attention is now being given to devices and materials which employ spin currents. This field, called “spintronics”, has been a topic of active research since the past three decades. A radical shift from the conventional electronic paradigm, devices based on spintronics have shown potential application in data storage, non-volatile magnetic random access memories (MRAMs)[1–3], spin based field effect transistors[4] etc. However, to interface spin based devices with charge based devices, we need to convert spin current into charge current. This can be made possible via inverse spin Hall effect (ISHE)[5, 6]. ISHE can be used to detect pure spin current generated by the phenomenon of spin pumping.

Spin pumping is the phenomenon of transfer of spin angular momentum from a ferromagnet (FM) to an adjacent non-magnetic (NM) material in the presence of an external magnetic field and an excitation microwave field. The precessing magnetic moments in the FM pump pure spin current into the NM layer which is subsequently dissipated by various relaxation processes. The magnitude of this spin current is maximum under the condition of ferromagnetic resonance. The spin current has the following form[7]:

$$J_s = \frac{\hbar}{4\pi} g_{eff}^{\uparrow\downarrow} \hat{m} \times \frac{d\hat{m}}{dt}$$

Here $g_{eff}^{\uparrow\downarrow}$ is the effective spin mixing conductance and $\hat{m}$ is the magnetization unit vector. If the NM has a high spin orbit coupling, then due to ISHE, the spin current may be detected electrically. As spin pumping opens up an additional channel of angular momentum dissipation, the intrinsic damping of the FM increases[8]. This increase is given as[8]:

$$\Delta \alpha = \frac{g_{\mu_B}^{\uparrow\downarrow}}{4\pi M_s d_{FM}} g_{eff}^{\uparrow\downarrow}$$

Here $\Delta \alpha$ is the difference between the Gilbert damping of the FM/NM bilayer and the single layer FM. $M_s$ is the saturation magnetization of the bilayer, $g$ is the gyromagnetic ratio, $\mu_B$ is the Bohr magneton and $d_{FM}$ is the ferromagnetic thickness. $g_{eff}^{\uparrow\downarrow}$ denotes the effective spin mixing conductance, an important parameter that characterizes the efficiency of transfer of spins through the FM/NM interface. However, damping may also increase due to inhomogeneities in the sample, interfacial defects, magnetic proximity effects, presence of capping layer[9, 10] etc. Therefore, only increase in damping cannot be taken as a sure indication of spin-pumping in a system. This is where electrical measurements for detection of spin pumping become necessary. ISHE stems from spin Orbit Coupling (SOC). SOC is a relativistic phenomena which couples the spin angular momentum to the orbital angular momentum of an electron. SOC is responsible for both spin Hall effect (SHE) and ISHE[11] i.e. it can convert charge current to spin current and vice versa respectively. As SOC is proportional to $Z^4$ (Z being the atomic number), usually heavy metals such as Pt[12], Pd[13], W[14], Ta[15] etc, are used for spin current detection via ISHE. However, it has been shown that 5d transition metal oxides, such as Iridium oxide, also exhibit high SOC, which originates due to the 5d electrons in the conduction band. The SOC is strong enough to reconstruct electronic structure as in the case of Sr$_2$IrO$_4$[16]. For example, the expectation value of the spin orbit coupling ($\langle L.S \rangle$) is $\approx 1.0\hbar^2$ for Ir, a heavy metal, while it is $\approx 3.1\hbar^2$ for IrO$_2$[17]. This shows that there is a signifi-

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cant SOC in Iridium oxide. Further, Fujiwara et. al. [18], have shown successful injection of spin current in IrO$_2$ wires in lateral spin valve geometry and have demonstrated ISHE with a remarkably high spin hall resistivity of around 38Ω-cm at room temperature. However, spin pumping via ferromagnetic resonance (FMR) in Iridium oxide/ Ferromagnet bilayer thin films has not been explored till now.

II. EXPERIMENTAL DETAILS

The thin films have been prepared on Si (100) substrates with native oxide, in a high vacuum chamber (manufactured by Mantis Deposition Ltd. UK) with base pressure better than 1.1 $\times$ 10$^{-7}$ mbar. We name the samples as S1, S2 and S3 for Si/Co$_{40}$Fe$_{40}$B$_{20}$(10), Si/Co$_{40}$Fe$_{40}$B$_{20}$(10)/IrO$_2$(2) and Si/Co$_{40}$Fe$_{40}$B$_{20}$(10)/Ir(3)/Cr(3), respectively. The numbers in brackets are in nm. Here S3 is the control sample. Co$_{40}$Fe$_{40}$B$_{20}$ and Ir were deposited via DC magnetron sputtering. For Co$_{40}$Fe$_{40}$B$_{20}$ the deposition pressure was $\approx$ 7 $\times$ 10$^{-4}$ mbar. The substrate was given a rotation of 20 rpm while deposition of CoFeB to have uniformity. The deposition rate was 0.21 Å/s at room temperature. For deposition of iridium oxide (sample S2), we followed the procedure as given by Liao et al. [19]. First, we increased the temperature of the substrate to 150$^\circ$C. Then we put oxygen gas into the chamber. The total pressure of Ar and O$_2$ was kept constant at 2.54 $\times$ 10$^{-3}$ mbar. Then Ir was DC sputtered from a 99.9% pure Ir target, at a rate of 0.16 Å/s onto the substrate. For Sample S3 CoFeB and Ir were DC sputtered while e-beam evaporation was used for Cr deposition. The deposition rates of Ir and CoFeB was 0.13 Å/s and 0.21 Å/s respectively with substrate rotation of 20 rpm. CoFeB was deposited at a temperature of 210$^\circ$C while Ir and Cr were deposited at 150$^\circ$C.

For x-ray diffraction (XRD) we have deposited a thicker IrO$_2$ film of 50 nm thickness. For samples S1-S3 we have also performed x-ray reflectivity (XRR) to calculate the thickness of the layers and the interface roughness. The XRD and XRR measurements have been performed with a Rigaku SmartLab diffractometer. For checking the Interface and crystalline quality we have performed cross-sectional transmission electron microscopy (TEM). Images were acquired by 300 kV TEM system of FEI, Tecnai G2 F30, S-Twin microscope, which was equipped with Gatan Orius CCD camera, HAADF detector, scanning unit and EDX spectrometer.

We performed frequency dependent FMR measurements (Phase FMR manufactured by NanoOsc, Sweden) on these samples to determine the intrinsic Gilbert damping. We used a modified FMR setup to detect ISHE in these samples the details of which are described elsewhere[20]. Further, we perform in-plane angular dependent measurement of the voltage signals with respect to the external magnetic field, to disentangle the contribution of spin pumping and other spin rectification effects in the obtained signal.

![FIG. 1. Experimental setup for determination of ISHE signal in the current sample structure Si/CFB(10)/IrO$_2$(2).](image)

A. Results and Discussion

For the purpose of determining whether we have actually obtained IrO$_2$ or not, we have performed XRD and TEM measurements. XRD was performed on a 50 nm single layer of IrO$_2$ on Si p-100 substrate. A weak peak was observed at around $\theta = 35^\circ$ corresponding to IrO$_2$(101) crystal[21].

![FIG. 2. XRD of 50nm IrO$_2$ single layer thin film. A weak peak is observed at $\sim 35^\circ$ which corresponds to the 101 crystal axis.](image)

We also performed Energy Dispersive X-Ray (EDX) and TEM on the samples to observe the growth of The various layers. The EDX measurements showed definite presence of Oxygen and iridium in the sample hinting significant formation of IrO$_2$. The ratio of Ir to Oxygen is
not 1:2, suggesting presence of pure Ir as well. The TEM images corroborated our claims by showing a formation of \( \sim 2 \text{ nm} \) thick layer of IrO\(_2\). There has been oxidation of CoFeB, as well as diffusion of Ir into the oxide layer, as evidenced by an extra layer in between the IrO\(_2\) and CoFeB layer.

For the purpose of measuring intrinsic Gilbert damping, we have used a Co-planar waveguide FMR (CPW-FMR) set up. The sample is placed face down on the waveguide, which transmits a radio frequency electromagnetic (EM) wave through it. The EM wave contains the perturbing field \( h_f \) which is perpendicular to the applied DC field used to saturate the magnetization. The CPW-FMR utilises the coupling between the rf wave and the precessing magnetization of the ferromagnet, and extracts the transmission coefficient of the waveguide. The rate of change of the transmission coefficient is maximum at resonance. The present device (NanoOSC Instruments PhaseFMR-40) uses a lock-in technique to detect this change, and thus, we record the derivative of absorption of the rf signal.

For the present experiment, in a single run, we have kept the frequency constant and varied the external field to obtain the resonance. This was performed over a range of frequencies starting from 4 GHz to 12 GHz, with an interval of 0.5 GHz for all the four samples. The obtained spectra was then fitted to a lorentzian function to obtain the Resonant field \((H_{\text{res}})\), and the linewidth \((\Delta H)\). From these information, we then extracted the damping values for the samples.

For measurement of ISHE, we used a slightly modified FMR setup. The detailed procedure is given in[20]. The sample size used for the purpose was a 2mm \( \times \) 3mm sample. The contacts (copper wires) were attached along the 2mm edge using silver paste. A nano-voltmeter was used to measure the voltage signal from the sample. Angle dependent measurement of the voltage signal with respect to the external field was performed for all the four samples to extract individually, the contribution of spin pumping and the various spin rectification effects.

The following function was used to fit the FMR spectra:

\[
I = 4A \frac{\Delta H(H - H_{\text{res}})}{(4(H - H_{\text{res}})^2 + H^2)^2} - S \frac{\Delta H^2 - 4(H - H_{\text{res}})^2}{(4(H - H_{\text{res}})^2 + H^2)^2}
\]

(3)

\( I \) is the FMR absorption derivative. \( A \) denotes the anti-symmetric and \( S \) denotes the Symmetric part of the lorentzian function. \( H \) is the applied field. We extract the values of \( H_{\text{res}} \) (the resonant field) and \( \Delta H \).

From the frequency-\(H_{\text{res}}\) variation, we perform the Kittel fitting[22]:

\[
f = \frac{\gamma}{2\pi} \sqrt{(H_{\text{res}} + H_k)(H_{\text{res}} + H_k + 4\pi M_{\text{eff}})}
\]

(4)

Here \( H_k \) is the uniaxial anisotropy field, \( \gamma \) is the gyromagnetic ratio and \( M_{\text{eff}} \) is the effective magnetization (in CGS units).

From the obtained value of \( \gamma \) from the Kittel fittings, we fit the linewidth(\(\Delta H\))- frequency plots to obtain the value of the Gilbert damping. The equation used is:

\[
\Delta H = \frac{4\pi \alpha}{\gamma} f + \Delta H_0
\]

(5)

Here, \( \alpha \) is the Gilbert damping constant, and \( \Delta H_0 \) is the inhomogeneous broadening.

From the damping fits (Figure 4b), we find that while the damping of S1 is \( \approx 0.0088 \pm 0.0002 \), the damping increases for samples S2 (0.0092\pm0.0002) and S3 (0.01496\pm0.0001). In addition, the inhomogeneous broadening in S2 is 3.6 Oe while that in S3 is 17.5 Oe. The values of damping and inhomogeneities indicate that there might be spin pumping occurring in S2 which may be detected as voltage signals. However, other effects such as magnetic proximity effect may also contribute to the increase in damping[23].

We perform electrical detection experiments on samples S2 and S3 in order to quantify spin pumping in the systems. We see ISHE signals in S2, but not in S3. Figure 5 shows the ISHE signal in S2 and the corresponding symmetric and anti-symmetric contributions to it. To further quantify the contribution of different spin rectification effects like anomalous Hall effect (AHE), anisotropic magnetoresistance (AMR), etc. from the spin pumping effect, we perform in-plane angle dependent measurement of the voltage signal at 5° intervals. The angle \( \Phi \) is defined as the angle between the \( h_f \) and the external magnetic field. While the pure ISHE signal is symmetric in nature, spin rectification effects from the ferromagnetic layer have both symmetric and anti-symmetric components [24–26]. Thus, one needs to separate the obtained signal into symmetric and anti-symmetric components. The voltage signal is fitted to the following function, which is simply a combination of symmetric and anti-symmetric lorentzian.

![Image of Diffraction pattern of Si/CoFeB(10)/IrO\(_2\)(2) and TEM image of the same. There is a clear layer of IrO\(_2\) as evidenced by an extra layer in between the IrO\(_2\) and CoFeB layer.](image-url)
FIG. 4. a) shows the Kittel fitting for all the samples. The red lines indicate best fits. b) shows the $\Delta H$ vs frequency dependence. The red lines indicate best fits.

$$V_{\text{meas}} = V_{\text{sym}} \left( \frac{(\Delta H)^2}{(H-H_{\text{Res}})^2 + (\Delta H)^2} \right) + V_{\text{asym}} \left( \frac{\Delta H (H-H_{\text{Res}})}{(H-H_{\text{Res}})^2 + (\Delta H)^2} \right) + C$$  \hspace{1cm} (6)

The green and cyan dotted lines in Figure 5 show the symmetric and anti-symmetric components respectively, while the red line shows the overall fit of the voltage signal.

For sample S2, we notice that there is a significant anti-symmetric component along with the symmetric component. This indicates a significant contribution of AHE and other anti-symmetric voltages in S2. However, spin rectification effects also have a symmetric component i.e the parallel and perpendicular AMR. Thus, we performed angle dependent analysis of the voltage signal in order to separate out these effects. The Landau-Lipshitz-Gilbert equation can be linearized in the small cone angle limit of the precessing magnetization. Thus, the measurements should be performed in the linear regime, which would otherwise have a large impact on the linewidth and Gilbert type damping will not be guaranteed[27]. Figure 6 shows linear variation of the anti-symmetric and symmetric voltages for all the samples. This is in accordance with the FMR spin pumping signal in the linear-response regime[28–30].

To explicitly quantify the contribution of spin pumping to the obtained voltage signal, we perform angle dependent analysis of the sample at 7 GHz. We vary the azimuthal angle $\Phi$ (defined previously) from 0° to 360° at an interval of 5° for both the samples. We then plot the symmetric and anti-symmetric components of both the samples with angle. Using the model developed by Harder[24], we then quantify the contribution of spin pumping and various spin rectification effects in the samples. We have assumed that the spin rectification comes from two sources: (Anomalous Hall Effect) AHE and (Anisotropic Magneto-Resistance) AMR. The equations used are:

$$V_{\text{Sym}} = V_{\text{sp}} \cos^3(\phi) + V_{\text{AHE}} \cos(\phi) \cos(\Phi) + V_{\text{AMR-\perp}} \cos(2\Phi) \cos(\Phi) + V_{\text{AMR-\parallel}} \sin(2\Phi) \cos(\Phi)$$ \hspace{1cm} (7)

$$V_{\text{Asym}} = V_{\text{AHE}} \sin(\phi) \cos(\Phi) + V_{\text{AMR-\perp}} \cos(2\Phi) \cos(\Phi) + V_{\text{AMR-\parallel}} \sin(2\Phi) \cos(\Phi)$$ \hspace{1cm} (8)

Here we $\phi$ is defined to be the phase angle between the...
electric and magnetic field of the microwave which is $90^\circ$. $\Phi$ is the angle between the $H_{rf}$ and the applied magnetic field.

From the fitted plots, we see that for sample S2, there is nearly equivalent contribution from both symmetric and anti-symmetric components. The spin pumping ($V_{SP}$) was found to be $2.79 \pm 0.09 \, \mu V$. The anomalous Hall effect (AHE) voltage ($V_{\text{AHE}}$) was $1.64 \pm 0.06 \, \mu V$. The AMR contribution is calculated as:

$$
V_{\text{AMR-}(\parallel, \perp)} = \sqrt{(V_{\text{AMR-}(\parallel, \perp)}^{\text{Sym}})^2 + (V_{\text{AMR-}(\parallel, \perp)}^{\text{Asym}})^2}.
$$

For sample S2, $V_{\text{AMR-}} = 0.23 \pm 0.10 \mu V$ and $V_{\text{AMR-}} = 1.2 \pm 0.08 \mu V$. The value of $V_{\text{AMR-}}$ is one order less than the $V_{\text{AHE}}$ or $V_{SP}$. The small non-zero value is due to the fact that our contacts ($\approx 2 \, \text{mm}$) deviate from the ideal point-like conditions which is assumed in the model. $V_{\text{AMR-}}$ has a significant contribution to the voltage signal. This might be due to presence of anisotropy in the FM layer. However, the model could satisfy the observed behavior satisfactorily with minimal deviations.

We have calculated the spin mixing conductance ($g_{eff}^{\uparrow \downarrow}$) using the following equation\cite{8, 29, 31}, in order to determine the efficiency of transfer of spins through the FM/NM interface.

$$
g_{eff}^{\uparrow \downarrow} = \frac{4\pi M_s d_{FM} \Delta \alpha}{g \mu_B}
$$

The saturation magnetization $M_s$ was measured by SQUID magnetometer. For S2, it was found to be $\approx 704 \, \text{emu/cc}$. The $g_{eff}$ thus becomes $0.188 \times 10^{19} \, \text{m}^{-2}$. To compare, the $g_{eff}$ for NiFe/CuIr sample ranges from $0.99 - 0.57 \times 10^{19} \, \text{m}^{-2}$ \cite{32}. The obtained $g_{eff}^{\uparrow \downarrow}$ values are comparatively lower.

We were further interested in calculating the spin hall angle ($\theta_{SHA}$) for the sample. $\theta_{SHA}$ is the figure of merit of any spintronic device and shows the ratio of conversion of spin current to charge current. High $\theta_{SHA}$ is better for spintronic applications. $\theta_{SHA}$ is related to the $V_{ISHE}$ by the following relation \cite{33}

$$
V_{ISHE} = \left( \frac{w}{\mu_0 \gamma t_{IrO_2} + \gamma \rho_{CFB}} \right) \times \theta_{SHA} \lambda_{SD} \tanh \left[ \frac{t_p}{2\lambda_{SD}} \right] J_s
$$

where $J_s$ is given as:

$$
J_s \approx \left( \frac{\lambda_{eff}^2 h}{8\pi} \right) \left( \frac{\mu_0 h_{rf} \gamma}{\alpha} \right)^2 \times
$$

$$
\left[ \frac{\mu_0 M_s c + \sqrt{\left(\mu_0 M_s c\right)^2 + 16(\pi f)^2}}{\left(\mu_0 M_s c\right)^2 + 16(\pi f)^2} \right] \left( \frac{2e}{h} \right)
$$

$g_{eff}^{\uparrow \downarrow}$ is the real part of $g_{eff}^{\uparrow \downarrow}$ (Equation 9) and is given as:

$$
g_{r}^{\uparrow \downarrow} = g_{eff}^{\uparrow \downarrow} \left[ 1 + \frac{g_{eff}^{\uparrow \downarrow} \rho_{IrO_2} \lambda_{IrO_2} e^2}{2\pi h \tanh \left( \frac{t_{IrO_2}}{\lambda_{IrO_2}} \right)} \right]^{-1}
$$

The $\theta_{SHA}$ is found to be $\sim 3.7$. To determine the efficiency of a material for its use as a spin detector, spin hall resistivity ($\rho_{SH}$) is an important parameter. Ideally,
materials with large spin hall resistivity act as better spin detectors. $\rho_{SH}$ is given as [18]

$$\rho_{SH} = \rho_C \theta_{SHA}$$  \hspace{1cm} (13)

In our case we have found the resistivity ($\rho_C$) of IrO$_2$ to be 36.5 $\mu\Omega$ cm. Thus $\rho_{SH} = 135.05 \mu\Omega cm$, which is one of the highest reported value till date. The spin hall conductivity $\sigma_{SH}$ thus becomes $7.4 \times 10^5 \Omega^{-1} m^{-1}$.

In addition to $\theta_{SHA}$, spin interface transparency is another important parameter to determine the efficiency of spintronic devices. The expression is given as [34]

$$T = \frac{g_r^{\downarrow} \tanh \left( \frac{t_{IrO_2}}{\lambda_{IrO_2}} \right)}{g_r^{\uparrow} \coth \left( \frac{t_{IrO_2}}{\lambda_{IrO_2}} \right) + \frac{h}{2\pi \rho_{IrO_2} t_{IrO_2}}}$$  \hspace{1cm} (14)

We have used the literature value of spin diffusion length of IrO$_2$ ($\lambda_{IrO_2}$) which is 3.8 nm for poly-crystalline IrO$_2$ [18]. The spin interface transparency is then found to be $0.05 \pm 0.001$.

III. CONCLUSION

To summarize, we have successfully fabricated samples of IrO$_2$/CoFeB bilayers and have confirmed the same by XRD and TEM measurements. The IrO$_2$ formed is poly-crystalline in nature which will be of practical importance for devices. For the first time, we have observed ISHE signals in thin films via FMR. We could get ISHE signals with r.f power as low as 6 mW. This shows that the present system is highly receptive to low magnitude of spin current. Occurrence of spin pumping was indicated by frequency dependent experiments and was subsequently quantified by in-plane angle dependent ISHE experiments. Spin pumping was shown to have the dominant contribution. We further determine the $\theta_{SHA}$ to be $\approx 3.7$ whereas the spin hall conductivity was determined to be $7.4 \times 10^5 \Omega^{-1} m^{-1}$. The very high value of $\theta_{SHA}$ may be due to back-scattering and reflection of spins at the interface. We also confirmed that in our detection set-up, presence of IrO$_2$ is necessary for obtaining ISHE signals. The experiment serves to indicate that iridium oxide may be a robust spin current detector and warrants further investigation for its applicability in devices and thin-films.

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