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ABSTRACT

We measured the local magneto-transport (MT) signal with an out-of-plane magnetic field, including magneto-resistance (MR) and Extraordinary Hall effect (EHE), in exchange-biased [Co/Pd]IrMn thin multilayers that are micro-structured with a 100 μm window. We found that when measured locally around the window, the MT signal deviate from the expected behavior. We studied possible causes, including film micro-structuration, electrical contact geometry as well as magnetic field angular tilt. We found that tilting the magnetic field direction with respect to the normal direction does not significantly affect the MT signal, whereas the positioning and geometry of the contacts seem to highly affect the MT signal. For comparison purposes, we carried these MT measurements using the Van-der-Pauw method on a set of four microscopic contacts directly surrounding the window, and on another set of micro-contacts located outside the window, as well as a set of four contacts positioned several millimeters away of each other at the corners of the wafer. If the contacts are sufficiently far apart, the EHE and MR signals have the expected shape and are not significantly affected by the presence of the window. If, on the other hand, the contacts are micro-positioned, the shape of the EHE signal is drastically deformed, and may be modeled as a mix of the standard EHE and MR signals measured on the outer contacts. Furthermore, if the micro-contacts are located directly around the window, the deformation is amplified, and the weight of the MR signal in the mix is further increased by about 40 %. This suggests that the electron path in the Hall geometry is disturbed by both the proximity of the electrodes and by the presence of the window, which both contribute to the deformation for about two-third and one third, respectively.

INTRODUCTION

Multilayered [Co/Pd]IrMn thin films, as sketched in Fig.1a, exhibit interesting magnetic properties.[1–3] One of these properties is exchange-bias (EB),[4] caused by interfacial couplings between the ferromagnetic (FM) Co/Pd multilayers and the antiferromagnetic (AF) IrMn layers, occurring when the film is field-cooled below its blocking temperature T_B.[5] We found in previous studies[6–8] carried on [[Co (4Å)/Pd (7Å)]$_{x12}$/IrMn(24Å)]$_{x4}$ for which $T_B \sim 275$ K, that these exchange couplings induce remarkable Magnetic Domain Memory (MDM). The observed MDM is the highest and the most extended throughout the magnetization loop when the cooling field is close to zero (remanence).[9] At remanence, the magnetic domains in the F layer tend to form a maze pattern, like the one illustrated in Fig. 1b, which gets imprinted in the AF layer upon cooling. We carried these studies using synchrotron x-ray radiation, which allows probing the domain pattern morphological changes at the nanoscale while applying a magnetic field in-situ.[10] However, recently, we observed an unexpected loss of MDM upon field cycling. We have been investigating possible reasons for the MDM loss, including x-ray illumination effects. For these investigations, we used in-situ magneto-transport (MT) while under x-ray illumination, to see if the MDM loss may be accompanied by a loss of EB. In practice, EB consists in the biasing of the magnetization loop with the respect to the applied magnetic field, in the direction opposite to the direction of the previously applied cooling field. Our earlier magnetization

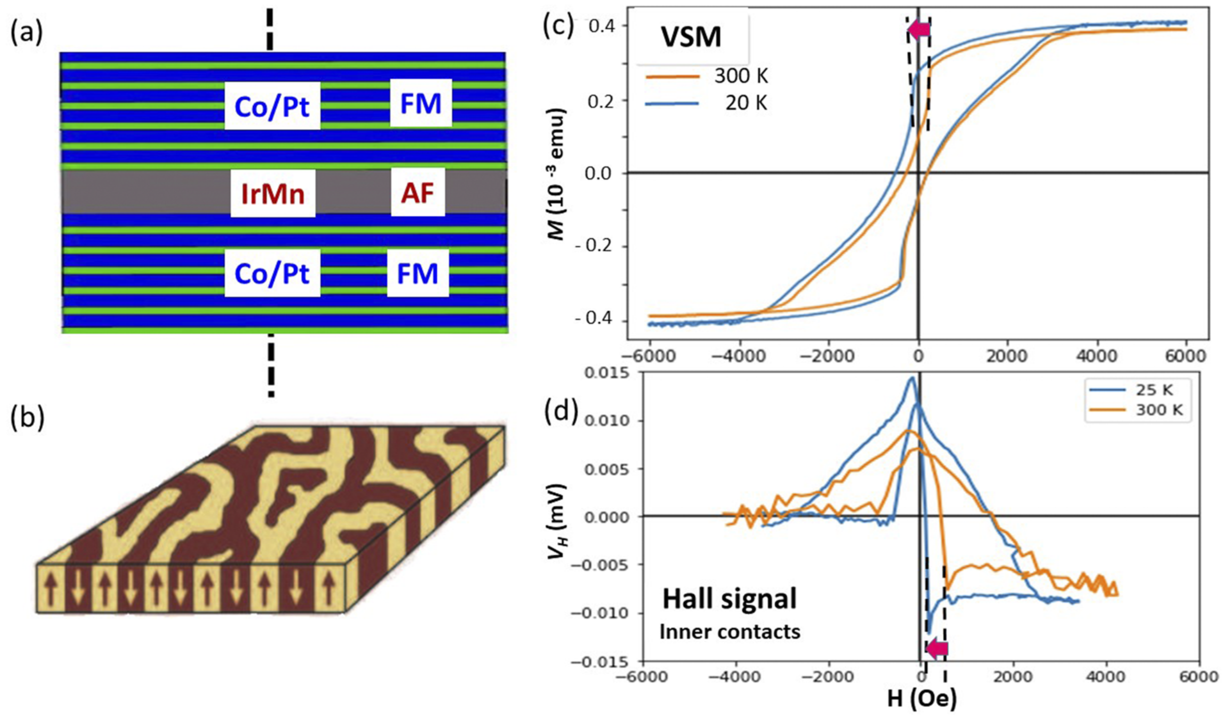

FIG. 1. (a) Sketch of the [[Co (4Å)/Pd (7Å)]$_{x12}$/IrMn(24Å)]$_{x4}$ multilayer structure; (b) Illustration of magnetic domain pattern forming in the film near remanence; (c) Magnetization loops measured via VSM at 300 K and at 20 K after field cooling under a + 6000 Oe field; (d) Magnetization loops measured via EHE under a current of 100 mA on inner contacts at 300 K and 25 K after field cooling under +4750 Oe. In both VSM and EHE measurements, the cooling field was well above saturation point H_s ~ 3200 Oe.

measurements on [[Co (4Å)/Pd (7Å)]$_{x12}$/IrMn(24Å)]$_{x4}$, shown in Fig.1c using Vibrating Sample Magnetometry (VSM), indicate a bias field as high as 200 Oe at 20 K. When measuring EB via MT, we could observe a biasing effect consistent with the VSM measurements. However, we found that the shape of the Extraordinary Hall Effect (EHE) signal (see Fig.1d) is deformed with respect to the expected magnetization loop shape. This paper investigates possible reasons for this deformation.

METHODOLOGY

The synchrotron x-ray magnetic scattering measurements were carried at the Advanced Photon Source, beamline 4-ID-C, in a vacuum chamber equipped with an in-situ octupole magnet. To allow x-ray scattering measurements in transmission geometry, the [[Co (4Å)/Pd (7Å)]$_{x12}$/IrMn(24Å)]$_{x4}$ thin films were deposited onto 100 nm thick Si_3N_4 membranes supported by silicon wafers that have a 100 μm window at their center. To enable MT measurements, the films were electrically hooked to a circuit board with ultra-thin 20 μm wires soldiered via wire-bonding, as seen in Fig.2a. The electrical contacts were grouped by sets of four, to enable both Extraordinary Hall Effect (EHE) and magneto-resistance (MR) measurements using the Van-der-Pauw method[11,12] with a magnetic field applied out-of-plane.

To study the effect of x-ray illumination on the exchange couplings and a possible loss of EB, the MT signal was measured locally, as close as possible to the illuminated window. For this purpose, we created four electrical contacts nearby the central window by depositing four Pt pads using Focused Ion Beam (FIB). The geometry and location of these four pads are shown in the sketch Fig.2b and in the Scanning Electron Microscopy (SEM) image Fig.2c. Each pad has a shape of a 125 x 125 μm^2 square, that is 100 nm thick. The four pads are diagonally located by the four corners of the window at a distance of 300 μm of each other. Along the diagonal, the distance between pads is about 425 μm center-to-center and the central window covers about 33% of that distance. Additional to these micrometric "inner" contacts located around the window (ON-window), we created, for comparison purposes, another set of four "inner" contacts with similar geometry but located outside the window (OFF-window), as well as a set of four "outer" contacts located by the four corners of the wafer at about 5 mm of each other, as schematically represented in Fig.2b.

When measuring the MT signal on the inner contacts while using the octupole magnetic chamber, we found that the EHE signal, shown in Fig.1d, was deformed with respect to the expected hysteresis loop, as measured via VSM (see Fig.1c). The EHE signal appeared like a folded hysteresis loop.

To investigate the origin of this deformation, we conducted a series of MT measurements in our laboratory at BYU. One investigation consisted in comparing the MT signal on the inner contacts to the MT signal on the outer contacts, used as a reference. Another investigation consisted in comparing the MT signal measured on the

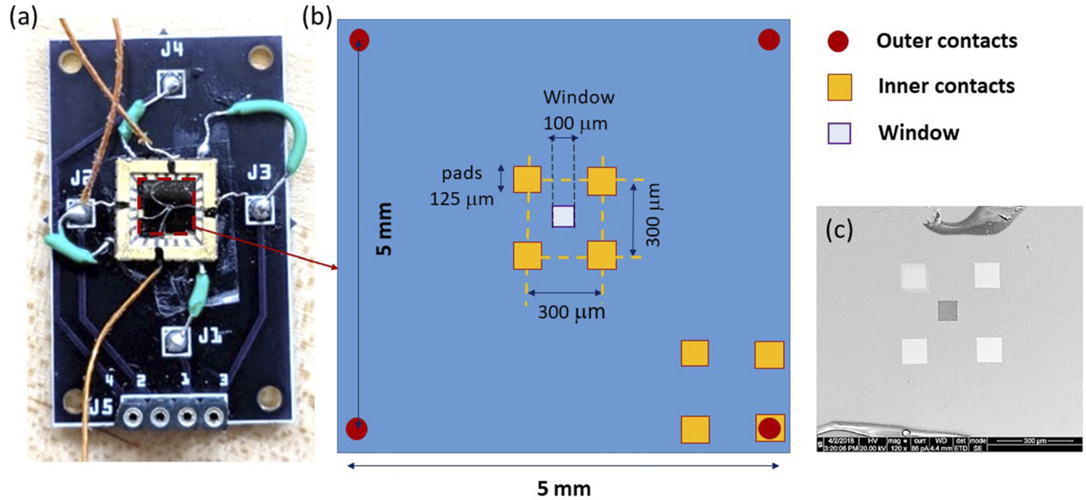

FIG. 2. (a) Picture of the electrical board on which the film is mounted and electrically connected via wire-bonding; (b) Sketch showing the location of the outer contacts at the four corners of the film and the location of the inner contacts surrounding the central 100 μm window; (c) SEM image of the padded inner contact deposited via FIB surrounding the 100 μm window.

inner contacts ON-window and OFF-window to identify possible effects caused by the window itself. Additionally, we studied possible effects caused by tilting the applied magnetic field with respect to the direction normal to the film surface. Indeed, during the synchrotron measurement, one of the eight poles of the octupole electromagnet failed, causing a tilting of the applied magnetic field with respect to the direction of the x-rays. In addition, due to space constraints, the sample holder was also tilted, causing a tilt of the film with respect

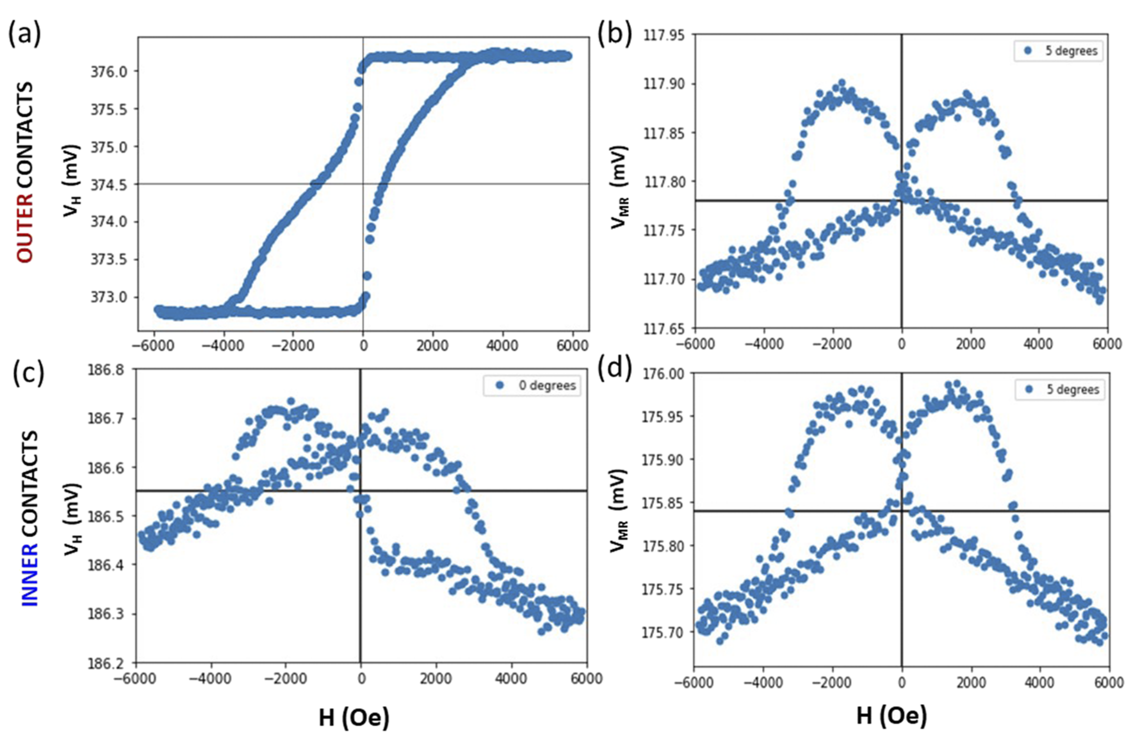

FIG. 3. (a,b) Actual MT signal measured on the ***outer contacts:*** (a) EHE signal and (b) MR signal; (c,d) Actual MT signal measured on the ON-window ***inner contacts***: (c) EHE signal and (d) MR signal. All the measurements were carried under a current of 100 mA.

to the x-ray direction. The combination of these two angular deviations resulted in a total tilt between the magnetic field direction and normal to the film surface up to about 25°.

The BYU MT measurements were carried in a bipolar electromagnet. The EHE signal, on one hand, was obtained by measuring the voltage in the transverse direction with respect to the applied current, while the magnetic field is applied perpendicular to the film. For the EHE data, averages between such transverse measurements measured at 90° of each other were taken. The MR signal, on the other hand, was obtained by measuring the voltage in the direction parallel to the applied current using the four contacts and applying the Van-der-Pauw method, where four different configurations were averaged to eliminate possible structural asymmetries.[12]

RESULTS AND DISCUSSION

The MT signal measured on the outer contacts, displayed in Fig.3 a,b, shows a behavior typical of ferromagnetic materials. The averaged EHE signal in Fig.3a has the shape of a hysteresis loop, consistent with the VSM signal. The averaged MR signal in Fig.3b has a symmetrical double-lobe shape typical of magneto-resistance in ferromagnetic thin films with perpendicular magnetic anisotropy.[13,14] The measured MR voltage is around 118 mV for a current of 100 mA, corresponding to a resistance of $R_{//} \approx 1.2\Omega$ with magneto-resistance variation $\Delta R \approx 210^{-3}\Omega$.

The MT signal measured on the ON-window inner contacts however behaves differently compared to the MT signal measured on the outer contacts. The measured EHE signal in Fig.3c has a deformed shape with respect to the expected hysteresis loop. The deformed shape looks like a hysteresis loop folded onto itself in an asymmetrical way, leading to two apparent hysteresis loops, a smaller one and a bigger one. On the other hand, the average MR signal in Fig.3d is similar to the MR signal measured on the outer contacts (Fig.3b), still showing a symmetrical double-lobe shape. The measured MR voltage is around 178 mV for a current of 100 mA, corresponding to a resistance $R_{//} \approx 1.8\Omega$, with magneto-resistance variation $\Delta R \approx 310^{-3}\Omega$.

The dependence with magnetic field tilt was studied by tilting the sample holder with respect to the electromagnet axis. The setup allowed a tilt up to 20°. Data collected at various angles is plotted in Fig.4 For comparison purposes, the data in Fig.4 was normalized to the maximum value (plotting $V/V_{\max}$) after recentering the signal, so that both the EHE and MR magnetization loop signal varies between − 1 and +1. The data shows no significant effect of magnetic field tilt on the shape of the EHE and MR signals, neither on the outer contacts nor on the inner contacts. This observation rules out any correlation between the observed EHE signal deformation and the actual magnetic field tilt during the synchrotron measurement.

It then appears that the deformation of the EHE signal observed on the inner contacts is principally due to micro-structuration

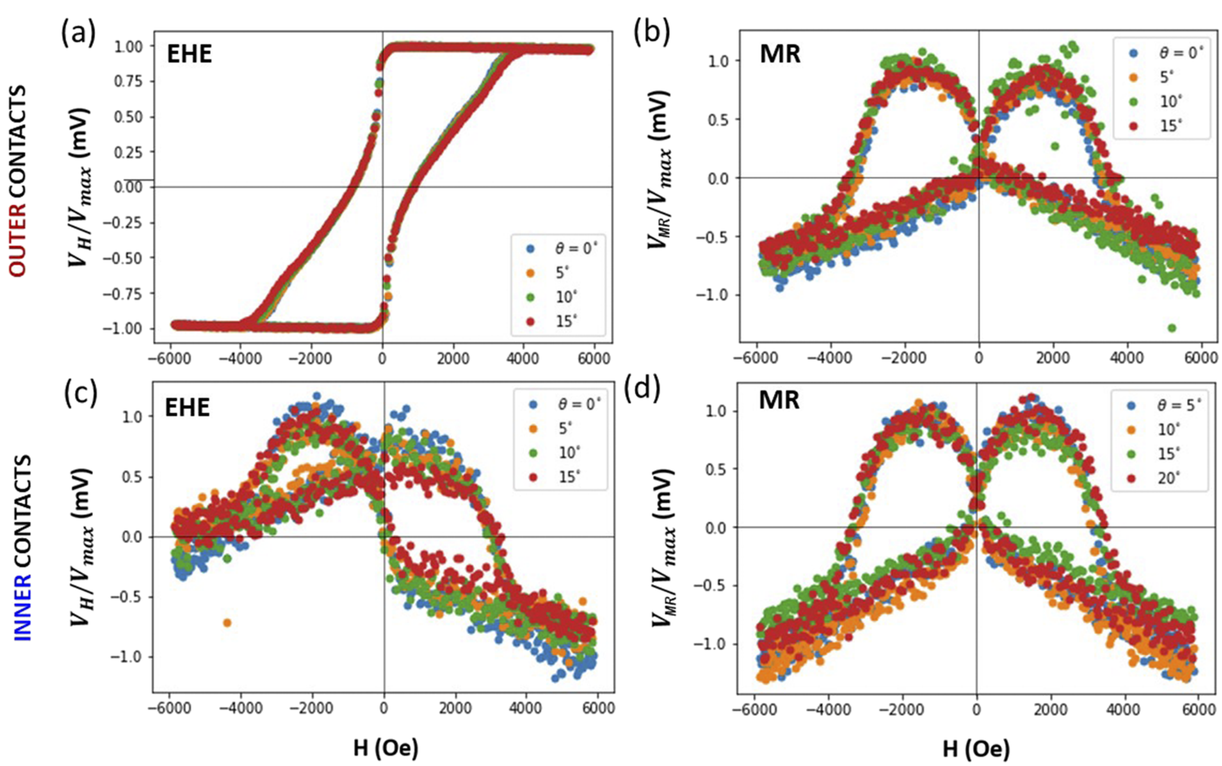

FIG. 4. (a,b) Normalized MT signal measured on the ***outer contacts:*** (a) EHE signal and (b) MR signal; (c,d) Normalized MT signal measured on the ON-window ***inner contacts***: (c) EHE signal and (d) MR signal. Measurements at various angles from 0 to 20° are displayed.

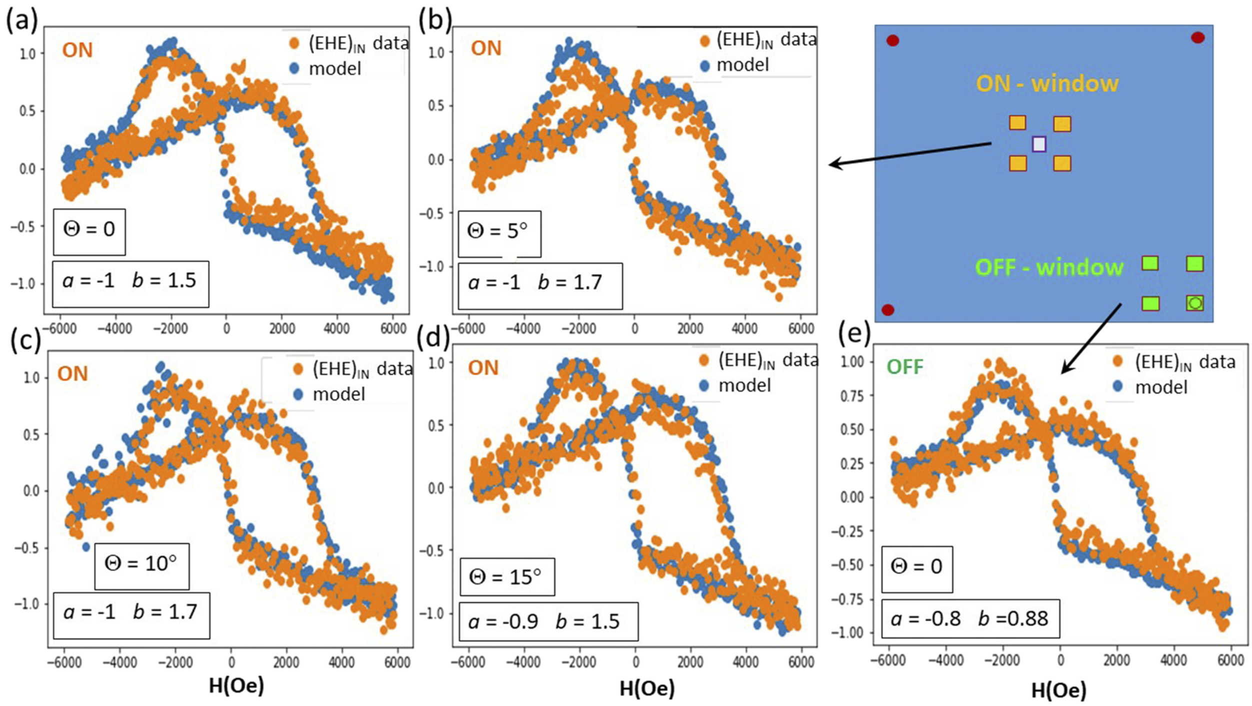

FIG. 5. Modeling of the EHE signal measured on two sets of inner contacts: (a-d) around the window (ON-window) and (e) outside the window (OFF-window) for comparison. The location of the sets of contacts is schematically represented on the diagram. The data was collected as follows: ON–window at an angle of (a) $\theta = 0$; (b) $\theta = 5^{\circ}$; (c) $\theta = 10^{\circ}$; (d) $\theta = 15^{\circ}$; (e) OFF–window at an angle of $\theta = 0$. For each data set, the model uses a linear combination of the normalized EHE and MR signals measured on the outer contacts, as follows: $(EHE)_{IN} = a * (EHE)_{OUT} + b * (MR)_{OUT}$.

(window) effects and geometry of the contacts. When measuring the MT signal using the inner contacts, the electron path is significantly disturbed by the presence of the window, which occupies 33% of the distance between electrodes, and also by the relative width of the pads (125 μm) with respect to the inter-pad distance (300 μm), that occupies about 45 % of that inter-electrode distance. This suggests that electrons may not travel on straight paths between diagonally opposite contacts, but instead may deviate from the straight path to get around the central window. In this process, some electrons may end up hitting adjacent contacts instead of opposite contacts, causing some mixing between EHE and MR signals.

To support this hypothesis, we attempted modeling the EHE signal measured on inner contacts by using a linear combination of the EHE and MR signals measured on the outer contacts as follows:

$$(EHE)_{IN} = a * (EHE)_{OUT} + b * (MR)_{OUT}$$

The modeling results displayed in Fig. 5 show that one can indeed reconstruct the asymmetric shape of $(EHE)_{IN}$ by mixing the $(EHE)_{OUT}$ and $(MR)_{OUT}$ signals with coefficients a and b in opposite signs. The ratio $|b/a|$ for the EHE signal for the ON-window inner contacts ranges between 1.5 and 1.7 for the various sets measured at different tilt angles.

To further disentangle possible separate effects caused by the window on one hand and by the contact geometry on the other hand, we measured the EHE signal on a set of inner contacts located OFF-window, as illustrated in Fig.5. The measured OFF-window EHE signal, shown in Fig.5e, has a shape similar to the ON-window EHE signal. The modeling of the OFF-window EHE signal leads to a ratio $|b/a| \approx 1.1$. The significant change in the $|b/a|$ ratio from about 1.1 up to 1.6 when moving from the OFF-window to ON-window contacts confirms that the window does contribute significantly to the deformation of the EHE signal. While the proximity of the contacts induces about 2/3 (~70%) of the deformation, the presence of the window induces another 1/3 (~30%) of the deformation. This suggests possible pulling effects at the edge of the window, which could be caused by misalignments between the plane of the film and the plane of the window, as well as morphological defects or discontinuities in the structure of the multilayered film at the edges of the window. These micro-structuration effects are therefore non-negligeable when measuring the MT signal locally and must be taken into consideration.

CONCLUSION

We have probed the local magneto-transport (MT) signal, including magneto-resistance (MR) and Extraordinary Hall Effect (EHE), in exchange-biased $[[Co\ (4Å)/Pd\ (7Å)]_{x12}/IrMn(24Å)]_{x4}$ thin films that are micro-structured with a central 100 μm window. We carried these measurements using the Van-der-Pauw method on three sets of four contacts: a set of outer contacts located 5 mm apart at the four corners of the film; a set of inner contacts, made of 125 μm pads located at 300 μm of each other, surrounding the central 100 μm window, and another set of inner contacts with same geometry, located outside the window toward the corner of the wafer, for comparison purposes. We found that when measured on the outer contacts, the MT signal has the expected shape, with the EHE signal forming a hysteresis loop consistent with magnetometry measurements, and the MR signal showing a symmetrical double-lobe shape. When measured on the inner contacts, the MR signal still shows the symmetrical double-lobe shape, however the EHE signal is significantly deformed, looking like an asymmetric folded hysteresis loop. This deformation was observed both with the ON-window contacts and the OFF-window contacts. The deformed $(\mathrm{EHE})_{\mathrm{IN}}$ shape may be reconstructed by mixing the $(\mathrm{EHE})_{\mathrm{OUT}}$ and $(\mathrm{MR})_{\mathrm{OUT}}$ signals measured on the outer contacts. The relative MR/EHE weight ratio was found to be in the range of 1.5 to 1.7 for the ON-window contacts, and around 1.1 for the OFF-window contacts. This suggests that when EHE is probed locally with electrodes at close proximity, the electrons are not traveling in a straight path between diagonally opposite contacts but a portion of them hit the adjacent contacts instead, leading to a mix of MR and EHE signals. Furthermore, the presence of the window increases the weight of the MR signal in the deformation (an additional 40%). This suggests that the window causes pulling effect due to morphological misalignments and defects occurring at the edges of the window. Additionally, we found that moderately tilting the magnetic field with respect to the film normal direction is not affecting the shape of the MT signal significantly. The observed deformation of the EHE signal is mainly due to the micro-structuration of the film and the proximity of the inner contacts with the central window covering 33% of the path between contacts.

In order to measure effects of x-ray illumination on exchange-bias, it is however necessary to probe the MT signal locally around the illuminated region of the film, which necessitates some micro-structuration. Our current setup induces a deformation of the EHE signal. A solution to this issue may be to etch the [Co/Pd]IrMn film in the shape of a cross around the central window so to guide the electrons path in the desired direction for the EHE measurement. That being said, it is interesting to note that, despite the observed deformation, the EHE signal measured on the inner contacts with the current setup still shows the biasing effect when the film is cooled from 300 K down to 20 K below the blocking temperature, consistent with magnetometry measurements. So, even when its shape is deformed due to micro-structuration, the EHE signal may be used to measure exchange bias and monitor its dependence with various parameters such as temperature or x-ray illumination.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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