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Ferromagnetic resonance investigation of physical origins of modification of the perpendicular magnetic anisotropy in Pd/Co layered films in the presence of hydrogen gas

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In our work, we study the physical origins of the hydrogen gas induced reduction of the strength of perpendicular magnetic anisotropy (PMA) at the interface of cobalt and palladium layers. To this end, we grow these films on flexible substrates and carry out ferromagnetic resonance (FMR) measurements in the presence of two different stimuli—hydrogen gas and elastic strain. Exposing the samples to H_2 results in a downshift of the FMR field. On the contrary, FMR measurements carried out in the presence of an externally applied predominantly tensile elastic stress show an up-shift in the resonance field consistent with negative values of the saturation magnetostriction coefficient for our samples. Qualitative analysis of these results demonstrates that the magneto-elastic contribution to the hydrogen-induced change in PMA is very small and is of the opposite sign to the electronic contribution related to the influence of hydrogen ions on the hybridisation of cobalt and palladium orbitals at the interface. *Published by AIP Publishing.*
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I. INTRODUCTION

The interface perpendicular magnetic anisotropy (PMA) is known to be induced at the interface between a ferromagnetic metal (FM) layer and a non-magnetic (NM) heavy-metal layer such as platinum or palladium (Pd) in a FM/NM bilayer or multilayer film.[1–3] This phenomenon has been the focus of an intense research for several decades. Several origins of contributions to the interface anisotropy have been identified; the most important are: (1) breaking the crystal symmetry at the interface, (2) interface alloying, and (3) the effect of magnetostriction.[4] The first two mechanisms are underlain by a direct effect of a heavy metal on the spin system of the ferromagnetic layer, whereby spin orbit coupling plays an important role. Hence, this effect is of electronic nature[5] and for this reason in the following we will term the two contributions "the electronic contribution." The contribution (3) is indirect–elastic strain at the interface mediates the effect of NM on the magnetism of the FM layer.[6] Obviously, if the FM layer is thin enough, the elastic strain originating from the presence of the interface can propagate across the whole thickness of the FM layer and induce perpendicular anisotropy all across the layer's bulk *via* the magneto-striction effect. We will term this contribution to PMA "magneto-elastic."

For completeness, one has to also mention one more possible contribution to PMA-interface roughness.[6] It is of magnetostatic nature which suggests that the presence of the NM metal on the other side of the interface should not matter, and the effect of the roughness should be obviously the same as of roughness of the surface of a single ferromagnetic

layer (i.e., not interfaced with an NM metal). For this reason, we will neglect this contribution in the discussion below.

Recently, several authors have shown that Pd/FM multilayer systems represent a very interesting model system for studying the physics behind the interface PMA.[7–14] This is because incorporation of hydrogen atoms into Pd leads to a reversible change in the PMA strength. Pd has a very high affinity to hydrogen gas (H_2)—it absorbs large amounts of the gas reversibly at atmospheric pressure and room temperature. This makes control of PMA in the Pd/MN system an easy task, as the change of the effective PMA field has been found to scale with the partial pressure of the hydrogen gas in the layered film's environment. Interestingly, it has been recently observed that breaking the continuity of the films through lateral nanopatterning strongly decreases the characteristic time for the transient process of the change in PMA due to sample exposure to H_2.[13,14]

One important question in PMA research is the interplay between the two contributions to it which we mentioned above. The experiment with the nanopatterned samples[14] may suggest that the magneto-elastic contribution may be important or even dominating, since breaking the continuity of the films may allow easier lateral expansion of the film while exposed to the H_2 atmosphere. However, bulk cobalt is known to possess a negative magnetostriction coefficient,[15] therefore one may expect that the resonance field shift due to the magneto-elastic contribution is positive, which is in contradiction to a negative one observed in all previous in-plane ferromagnetic resonance (FMR) measurements in the presence of hydrogen gas.[11,14,16]

In this work, to elucidate the physics behind the change in the strength of PMA in the presence of hydrogen gas for Pd/FM layered films, we grow continuous Pd/cobalt bi-layer and Pd/Co/Pd tri-layer films on flexible Kapton® (Kpt) substrates

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(Kpt/film) and expose the grown films to hydrogen gas or elastic stress. Ferromagnetic resonance (FMR) measurements are taken in the presence of those stimuli. Exposing the samples to H_2 results in a downshift of the FMR field for both substrate types. On the contrary, FMR measurements carried out in the presence of an externally applied predominantly tensile elastic stress show an up-shift in the field consistent with negative values of the saturation magnetostriction coefficients for our samples. We carry out quantitative analysis of these data and find that the magnetostriction contribution to the H_2-induced FMR peak shift does not exceed 0.3 Oe for our films grown on Kpt and is much smaller than and of the opposite sign to the electronic contribution (20 to 30 Oe).

II. EXPERIMENT

In-plane continuous polycrystalline bi-layers Co(5 nm)/Pd(10 nm) and tri-layers Pd(20 nm)/Co(10 nm)/Pd(20 nm) were deposited with d.c. magnetron sputtering under an argon atmosphere. Previously, these two systems showed quite different responses to the presence of hydrogen gas,[16] potentially due to difference in textures of the cobalt and the capping Pd layers. Therefore it is worth investigating both systems.

The base pressure of the sputter system was below 5×10^{-8} Torr. The film sputtering was repeated twice in order to produce two batches of Pd/Co and two of Pd/Co/Pd films on Kpt substrates. This was done in order to be able to collect enough statistics.

Absorption of hydrogen gas by Pd layers is not a fully reversible process. Thicker (30 nm+) Pd layers clamped to substrates can develop folds, cracks, and even peel off.[17] Thinner films (as in our case) can withstand much larger deformations,[18] however for both thicker and thinner films, one may also expect an effect of hydrogen annealing[19] which may modify the crystal structure of Pd. Our previous studies showed that some irreversible changes to our films can indeed occur during their first exposure to hydrogen gas, especially diluted H_2 (see e.g., Fig. 5 in Ref. 20) These changes are usually relatively small and insignificant for the purpose of this work. However, because they are present, the FMR in the presence of H_2 must be regarded as a destructive experimental method (at least as far as the exposure of a virgin sample to H_2 is concerned). The FMR under elastic stress is a destructive method too. Therefore, care was taken in order to minimize the effect of irreversible changes on the experimental outcomes. To this end, the films from the same batch were sputtered simultaneously, in order to ensure that they are fully identical, and measurements with both methods were taken for samples in their virgin states.

One Kpt//film sample from each batch was characterised with an FMR-under-stress setup.[21,22] To enable this, the sample was glued to a piezoelectric actuator and a dc voltage was applied to the actuator to strain the sample. The second film of each batch was used to take measurements of the FMR response first in the presence of pure nitrogen gas (N_2) and then in the presence of pure hydrogen gas.

For both types of measurements, the broadband stripline field-modulated FMR method[23] was used. Microwave microstrip lines were utilised in order to couple microwaves to

magnetisation dynamics in the samples and to read samples' FMR responses. A small-amplitude ac magnetic field was applied to a sample in parallel to a large static magnetic field. The fields were oriented in the sample plane and along the stripline. A lock-in amplifier locked to the ac field frequency was employed to detect the FMR absorption signal. During the measurements, the microwave frequency was kept constant while the static magnetic field was swept linearly. The measurements under stress were carried out for several frequencies (6–12 GHz) while measurements under exposure to H_2 were performed at 10 GHz. Further details of the FMR-under-stress method can be found in Ref. 22 and of the FMR measurements in the presence of H_2 in Ref. 11.

Typical examples of the obtained raw FMR traces are shown in Fig. 1. One sees that application of elastic strain shifts the FMR field upwards [Fig. 1(a)], whereas exposure of the sample to H_2 leads to an FMR field downshift [Fig. 1(b)]. Previously we interpreted this downshift as a decrease in PMA.[11,16]

The last experiment which we carried out was observation of film bending in the presence of hydrogen gas. To this end, two extra films were grown on 21 mm-long and 2 mm-wide Kapton substrates. One of them was a Co(5 nm)/Pd(10 nm) bi-layer film and the second one a tri-layer Pd(20 nm)/Co(10 nm)/Pd(20 nm) film. They were placed in a transparent pressure-tight container which was filled either with pure hydrogen or air. The short (i.e., the 2 mm-long) edges of the strip were oriented vertically in order to avoid the effect of gravity on the measurement result. One short edge was fixed with a clamp and the other one was free to move. The position of the free edge was monitored with a microscope. We found that in the presence of hydrogen gas, the edge of the Pd/Co bi-layer sample moves by 59% of the substrate thickness and one of the Pd/Co/Pd tri-layer film by 157%. The shift is in the direction of the sample's substrate which is consistent with the elongation of the Pd layer in the presence of hydrogen and the known fact that palladium expands upon absorption of hydrogen gas.[24]

III. DISCUSSION

A noticeable in-plane uniaxial anisotropy H_u is present for the samples grown on Kpt substrates [Fig. 2(a)]. We
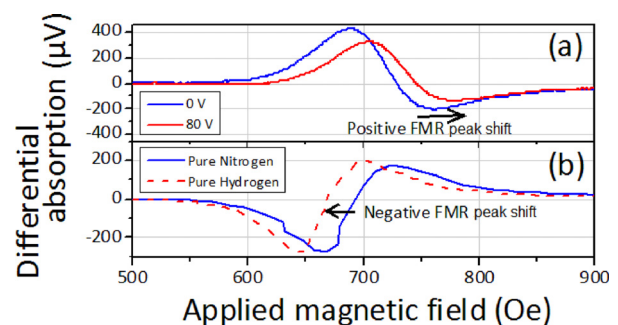

FIG. 1. Examples of raw FMR traces taken for a Kpt//Pd/Co/Pd sample. (a) FMR-under-stress, frequency is 8 GHz. Blue line: voltage applied to the piezo-actuator $V = 0$. Red line $V = 100$ V. (b) FMR in the presence of H_2. Blue solid line: taken in the atmosphere of pure N_2. Red dashed line: the atmosphere is pure H_2. Frequency for (b) is 10 GHz.
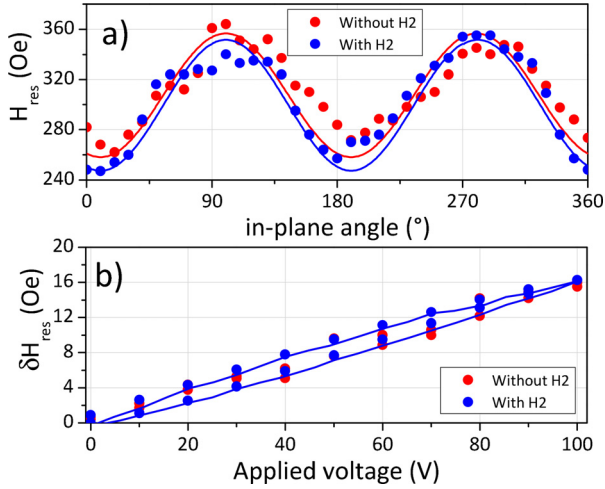
FIG. 2. (a) Results of the in-plane angle resolved FMR measurements for the same sample as in Fig. 1(a). (b) FMR field shift as a function of the voltage V applied to the piezo-actuator. "Without H2": virgin samples. "With H2": additional measurements after absorption and full desorption of hydrogen gas by the samples.

explain it as due to a potential bending of the substrate during sputtering.[25] Indeed, positioning of the sample on the stripline for taking the FMR measurements modifies the substrate's curvature. This leads to the anisotropy through magnetostriction. The FMR field shift due to application of a voltage V to the piezo-actuator is slightly non-linear due the in-plane strain (ε_{xx} and ε_{yy}) variations with voltage [see Fig. 2(b)].

From the FMR measurements taken for the Kpt//film samples in their virgin states, the samples' magnetic parameters were determined (Table I). The Co layers used in our study are thin enough such that the effective anisotropy field present at the interface does not need to be considered as leading to interface pinning of magnetisation;[26] instead one can use a simpler approach whereby the effective interface anisotropy field is replaced with an effective field of bulk PMA for the FM layer.[27] (In our previous work,[16] we found that the effective field of bulk PMA scales as 1/thickness for our bi-layer films (grown on Si substrates) which confirms the validity of the effective bulk anisotropy approach[27] for the Co layer thicknesses we deal with.)

Using the method from Ref. 21, values of the saturation magnetostriction coefficients for the materials were extracted from the results of samples' FMR characterisation under stress. These parameters are also listed in Table I.

TABLE I. Parameters of the samples extracted from the FMR measurements. Parameters of cobalt which were fixed to produce the results in the table are as follows: saturation magnetisation: 17.5 kOe, gyromagnetic ratio: 3.4 MHz/Oe, and Young modulus and Poisson ratio: 200 GPa and 0.3, respectively.

Samples	Description	PMA constant K_{perp} (Merg cm^{-3})	Saturation magnetostriction coefficient $\lambda/10^{-6}$	Hydrogen-induced FMR peak shift (Oe)
Batch 1	Kpt//Co/Pd	6.9	−16.0	−33
Batch 2	Kpt//Co/Pd	7.6	−13.5	−30.2
Batch 3	Kpt//Pd/Co/Pd	5.2	−3.2	−18.6
Batch 4	Kpt//Pd/Co/Pd	5.1	−3.0	−21

From the table, one sees that the saturation magnetostriction coefficient for our films has a negative sign as typical for cobalt.[15] Furthermore, we used the observed magnitude of bending of the long reference films in the presence of H$_2$ in order to extract the elastic strain ε_{H_2} in the cobalt layer induced by expansion of the Pd one. We employed formalism based on Ref. 28 to carry out this calculation. Its details are given in the Appendix. We obtained $\varepsilon_{H_2} = 2.8 \times 10^{-5}$ and 2.3×10^{-4} for the bi-layer and tri-layer film, respectively. By using the parameters from Table I, the strain values were converted into the magnetostriction contribution δH_{PMA} to the PMA field. To this end we employed the following expression:

$$\delta H_{PMA} = 3\lambda E_{Co}\varepsilon_{H_2}/[\mu_0(\nu - 1)M_s], \quad (1)$$

where λ is the saturation magnetostriction coefficient, $Y_{Co} = 200\,\text{GPa}$ and $\nu = 0.3$ are the Young modulus and Poisson ratio, respectively for cobalt, M_s is the saturation magnetisation for the cobalt layer, and μ_0 is the magnetic permeability of vacuum. The obtained change in the PMA field was further converted into the magnetostriction contribution to the H$_2$-induced change in the FMR field by using the Kittel equation for the in-plane FMR. It follows from the Kittel equation that the ferromagnetic resonance field reads

$$H_{res} = \left(\sqrt{(M_s - H_{PMA} + H_u)^2 + (2f/\gamma)^2} \right.$$
$$\left. -M_s + H_{PMA} + H_u \right)/2, \quad (2)$$

where H_{PMA} and H_u are effective fields of PMA and the in-plane uniaxial anisotropy respectively, f is the microwave frequency, and $\gamma = 3.4\,\text{MHz/Oe}$ is the gyromagnetic ratio.

We obtained $+0.27\,\text{Oe}$ and $+0.31\,\text{Oe}$ for the bi-layer film of Batch 2 and the tri-layer of Batch 3, respectively. One sees that these values are positive, in agreement with the negative values of λ from Table I and also negligible with respect to the total H$_2$-induced field shifts (last column of Table I).

IV. CONCLUSION

By combining FMR measurements under two different stimuli, we acquired experimental evidence that absorption of hydrogen gas by the Pd layer of Pd/Co and Pd/Co/Pd layered films results in modification of the electronic properties of the interface between the materials which leads to a change in the interface PMA strength. This evidence was obtained through exclusion of the possibility of the competing effect of magnetostriction to noticeably contribute to the hydrogen gas induced FMR peak shift. The electronic contribution to the change in PMA is negative, resulting in a negative in-plane FMR field shift.

It is known[29] that alloying of Co and Pd takes place at the interface. It was found experimentally that electronic hybridization of Co atoms near Pd neighbours is a PMA precursor for both Pd/Co layered films and PdCo alloys.[30] Palladium has three-times stronger spin-orbit coupling than cobalt, therefore it is likely that Pd enhances Co spin-orbit

coupling.[31] Absorption of hydrogen gas by alloys affects the magnetism of ferromagnetic-metal/Pd alloys.[32,33] It has been suggested that formation of Pd-H and Co-H chemical bonds reduces the Co-Pd bond overlap population.[32] This may reduce the effect of Pd atoms on Co spin-orbit coupling thus reducing the interface PMA. Hence, interface alloying may be an important condition allowing a significant change in the electronic properties of the interface in the presence of H_2.

On top of the electronic contribution, there may be a magneto-elastic (magnetostriction) contribution to the FMR peak shift. A free-standing piece of palladium expands uniformly in all three spatial directions when it absorbs hydrogen. If the palladium sample is a film clamped to a substrate, it cannot easily expand in its plane and a compressive bi-axial stress is induced in the sample. One may think of the stress as a hydrostatic pressure built-up in the sample. For thick Pd layers, the stress relaxes through formation of folds.[17] Thin films, as in our case, can withstand huge stresses due to the clamping effect without deformation.[18]

In contrast to single-layer Pd films used in Refs. 17 and 18, our films represent multilayers. Only the overlaying Pd layer of the tri-layer structures is able to absorb hydrogen, as cobalt is non-transparent to the gas. Hence, the layer which is capable of expanding under the influence of H_2 is separated from the substrate either by a cobalt layer or by a combination of Co and Pd layers in our case. However, these layers do not contribute to the mechanics, because of their negligible thickness with respect to the thickness of the substrate. Only the elastic properties of the capping Pd layer and the substrate matter, and the role of the other two metallic layers is just to translate the stress built-up in the capping Pd layer to the substrate in full.

Our experiment demonstrates that the magneto-elastic contribution to the change in PMA exists for samples grown on Kapton, but it is very small and is of the opposite sign to the electronic contribution. It arises because the flexible Kapton substrates can bend noticeably under the influence of the stress induced in the palladium layer by incorporation of hydrogen into the Pd lattice.

In all our previous studies,[11,14,16,34] we used samples grown on much more rigid silicon substrates. Because of the much larger rigidity of silicon (Young modulus > 130 GPa), bending of those samples due to the H_2-induced stress in the capping Pd layer is negligible, as follows from the value of the free-standing H_2-induced stress for the Pd layer found in the present work (see the Appendix). Accordingly, the magnetostriction contribution to the FMR peak shift for Pd/Co and Pd/Co/Pd films grown on silicon substrates completely vanishes.

This finding may suggest that nanopatterning-induced modification of magneto-elastic effects should be excluded as a potential reason for the recently observed drastic improvement in the time of response of nanopatterned Si//Pd/Co/Pd films on their exposure to hydrogen gas.[14]

ACKNOWLEDGMENTS

APPENDIX: EVALUATION OF THE STRAIN IN THE COBALT LAYER BASED ON THE MAGNITUDE OF SAMPLE BENDING IN THE PRESENCE OF HYDROGEN GAS

From the theory of bi-metal plates, it is known that a difference in strains in thin layers which are in a mechanical contact with each other leads to either spherical or cylindrical bending of the layered structure, depending on the stiffness of the materials (see e.g., Ref. 28). Ferromagnetic films on Kapton substrates are characterised by cylindrical bending (see Fig. 3 in Ref. 25). Furthermore, in our bending experiment we use a sample which is quasi-one-dimensional—a long and narrow (21×2 mm^2) strip. Therefore, in the following we utilise the model of bending into a cyllindrical roll to evaluate the strain.

The samples are fixed at one edge with the second edge free to move upwards or downwards. Assume that the sample is originally flat and its plane is parallel to the horizontal axis. Also assume that the Kpt subtstrate is facing up. This implies that elastic expansion of the Pd layer will result in the free edge of the strip moving up. The uplift h of the edge with respect to the horizontal axis relates to the raduis of bending R in the following way:

$$R = l^2/(2h), \qquad (A1)$$

where l is the strip length. The theory of tri-metals[35] tells us that the effect of the Co and Pd layers sandwiched between the capping Pd layer and the substrate on bending is negligible. This is because of smallness of the thicknesses of these burried layers with respect to the Kpt substrate thickness ($d_{Kpt} = 0.127$ mm), and is seen from the fact that under this condition the formula from that paper [Eq. (A4) in it] for the bending radius reduces to a very simple equation into which parameters of the burried layers do not enter

$$R = \frac{Y_{Kpt}d_{Kpt}^2}{6\varepsilon Y_{Pd}d_{Pd}}. \qquad (A2)$$

Here, $Y_{Kpt} = 4$ GPa and $Y_{Pd} = 120$ GPa are Young moduli for Kpt and Pd, respectively, $d_{Pd} = 10$ nm is the thickness of the capping Pd layer, and ε is the "free-standing" strain for Pd. The free-standing strain is the elastic strain which would be induced in the Pd layer if the layer was free-standing, i.e., not interfaced with the underlying Co layer and hence not

mechanically coupled to the remainder of the layered structure. The actual strain in the Pd layer is smaller than ε because the Kpt substrate does not allow the Pd layer to expand freely.

Equation (A2) is actually the Stoney formula for bi-metals (see e.g., Ref. 28). This implies that we may use the theory for bi-layered materials to evaluate the actual strain in the capping Pd layer. The strain in the buried Co layer will be the same as in the capping Pd layer, given the similarity of the Young modulus for Co (200 GPa) to the one of Pd and its strong dissimilarity from the one for Kpt.

It is appropriate to assume that there is no free-standing strain in Kpt ($e = 0$ for Kpt). Let us also assume that the axis in the direction of the bending radius is z, and $z = 0$ coincides with the free surface of the Kpt substrate. Then the free-standing strain in the stack has a profile as follows:

$$\begin{cases} e = 0, & 0 < z < d_{Kpt} \\ e = \varepsilon, & d_{Kpt} < z < d_{Kpt} + d_{Pd}. \end{cases} \quad (A3)$$

Given (A3) and under the condition $d_{Kpt} \gg d_{Pd}$, the formulas (4)–(6) from Ref. 28 reduce to a simple expression for the actual strain in the bending direction in the capping Pd layer ($\varepsilon_B(z = d_{Kpt} + d_{Pd}) \equiv \varepsilon_{act}$)

$$\varepsilon_{act} = \frac{d_{Kpt}}{R} \frac{3\nu_{Pd}(\nu_{Kpt} - 2\nu_{Pd} - 1) + 2(\nu_{Kpt} + 2)}{6(\nu_{Kpt} + 1)(\nu_{Kpt} - 2\nu_{Pd} + 1)}, \quad (A4)$$

where $\nu_{Pd} = 0.39$ and $\nu_{Kpt} = 0.34$ are the Poisson ratios for Pd and Kpt, respectively. As ε_{act} is the same for the Co layer, this formula can be used to evaluate ε_{act} in Co, based on the experimentally measured value of R.

The respective free-standing strain in the capping Pd layer is given by the expression as follows:

$$\varepsilon = \frac{Y_{Kpt} 3\nu_{Pd}(\nu_{Pd} - 1) d_{Kpt}^2}{6 R d_{Pd} Y_{pd}(\nu_{Pd} + 1)(\nu_{Kpt} - 2\nu_{Pd} + 1)} \quad (A5)$$

[which reduces to Eq. (A2) in the limit $\nu_{Kpt} = \nu_{Pd}$].

Importantly, the ferromagnetic films on Kpt develop a strain during their growth (see Fig. 3 in Ref. 25). For our strips, this natural cylindrical bending is visible with the naked eye. The radius of natural bending R_0 is easily found by measuring the elevation h_0 of the strip edge before H_2 has been let into the chamber and using Eq. (A1) to convert the elevation into R_0. Then the actual strain due to H_2 absorption reads

$$\varepsilon_{H_2} = \varepsilon_{act}(R_{H_2}) - \varepsilon_{act}(R_0), \quad (A6)$$

where $R_{H_2} = l^2 / [2(h_0 + \delta h)]$ is the radius of strip bending in the H_2 atmosphere and δh is the change in the elevation of the edge when the atmosphere in the chamber is changed to hydrogen gas from the original one (air in our experiment).

The original elevation of the edge of the 21 mm-long Kpt//Co/Pd strip was 0.9 mm. The uplift due to absorption of hydrogen $\delta h = 0.59 d_{Kpt} = 75 \, \mu m$. This yields a H_2 induced free-standing strain in Pd $\varepsilon = 2.4 \times 10^{-3}$. This value translates into $\varepsilon_{H_2} = 2.8 \times 10^{-5}$ which corresponds to expansion of the Co layer of the strip by 0.6 μm. Similarly, for the Kpt//Pd/Co/Pd strip our measurements yielded $h_0 = 0.58 \, mm$, $\delta h = 1.57 d_{Kpt} = 199 \, \mu m$. Interestingly, in its virgin state this

strip was bent in the opposite direction with respect to the bending direction due to H_2 absorption. In other words, originally the metallic layers were compressed. Absorption of H_2 reduced the negative strain and increased the bending radius. This has to be taken into account by changing the sign in front of δh in the expression for R_{H_2} (now $R_{H_2} = l^2 / [2(h_0 - \delta h)]$). The whole procedure yields $\varepsilon = 2 \times 10^{-2}$ and $\varepsilon_{H_2} = 2.3 \times 10^{-4}$. The latter value corresponds to Co layer elongation of 4.8 μm.

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