Magneto-ionic control of magnetism using a solid-state proton pump

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Voltage-gated ion transport as a means of manipulating magnetism electrically could enable ultralow-power memory, logic and sensor technologies. Earlier work made use of electric-field-driven O²⁻ displacement to modulate magnetism in thin films by controlling interfacial or bulk oxidation states. However, elevated temperatures are required and chemical and structural changes lead to irreversibility and device degradation. Here we show reversible and non-destructive toggling of magnetic anisotropy at room temperature using a small gate voltage through H⁺ pumping in all-solid-state heterostructures. We achieve 90° magnetization switching by H⁺ insertion at a Co/GdOₓ interface, with no degradation in magnetic properties after >2,000 cycles. We then demonstrate reversible anisotropy gating by hydrogen loading in Pd/Co/Pd heterostructures, making metal–metal interfaces susceptible to voltage control. The hydrogen storage metals Pd and Pt are high spin–orbit coupling materials commonly used to generate perpendicular magnetic anisotropy, Dzyaloshinskii–Moriya interaction, and spin–orbit torques in ferromagnet/heavy-metal heterostructures. Thus, our work provides a platform for voltage-controlled spin–orbitronics.

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Figure 1d–h shows out-of-plane hysteresis loops probed through the AHE resistance ($R_{\text{AHE}}$) for the initially oxidized sample. Results are shown for the virgin state (Fig. 1d) and after applying $V_G = +3 \text{ V}$ for 1,000 s at room temperature under various atmospheres (Fig. 1e–h). Consistent with the literature, under ambient atmosphere, a positive bias results in the appearance of out-of-plane magnetization, corresponding to the reduction of nonmagnetic CoO to metallic Co with perpendicular magnetic anisotropy (PMA) (Fig. 1e). Surprisingly, $V_G$ has no effect under vacuum (Fig. 1f), even though a lower-$p_T$ environment should make oxygen extraction more favourable. Likewise, in dry O$_2$ (Fig. 1g), no magnetic changes are observed, but remarkably, under wet N$_2$ (Fig. 1h), positive $V_G$ leads to the appearance of PMA, implying water-assisted reduction of CoO$_2$ to Co.

Figure 1i–m shows corresponding results for voltage-gated Co oxidation. Under ambient conditions, the AHE signal in the Co layer (Fig. 1i) vanishes after applying $V_G = -3 \text{ V}$ for 1,000 s (Fig. 1j). Since there is no corresponding onset of a PHE signal (not shown), which would be present if the magnetization simply reoriented in-plane, we infer that the Co is oxidized to form non-magnetic CoO$_2$. Under vacuum, voltage gating has no effect (Fig. 1k), which taken alone would suggest that oxidation occurs through O$^{2-}$ pumping from an atmospheric reservoir. However, under a pure O$_2$ environment (Fig. 1l), voltage-driven oxidation is slower than in ambient, whereas in oxygen-free wet N$_2$ (Fig. 1m) it is faster.

These results imply that the widely assumed mechanism of magneto-ionic switching through electric-field-driven oxygen displacement is incomplete, and that H$_2$O plays an essential role. It is well known that the water splitting reaction can be catalysed by noble metals with oxide support. Water splitting and hydrogen incorporation have been shown to substantially affect the switching behaviour and electronic properties in metal/oxide/metal memristors. Similar reactions in the present Pt/Co/GdO$_x$/Au stacks would provide a simple, self-consistent explanation of the results in Fig. 1.

During reduction of CoO$_2$ under positive $V_G$, we propose that H$_2$O is hydrolysed at the top electrode, producing H$^+$ and O$_2$ through the oxygen evolution reaction. The proton is transported to the bottom electrode via a Grothuss-type mechanism, in which the H$^+$ ion hops from an OH$^-$ to the adjacent O$^{2-}$ ion, enabled by solid-state dissolution of water in the oxide matrix (see Supplementary Information section 3). At the bottom electrode, the proton reacts with CoO to form Co and H$_2$O. The net reactions, depicted in Fig. 2a, are:

$$\text{Anode (Au):} \quad 2\text{H}_2\text{O} \rightarrow 4\text{H}^++\text{O}_2+4e^- \quad (1)$$
The water evolved at the bottom electrode can either be stored in grain boundaries, or is more likely to be incorporated into GdOₓ itself as hydroxide, namely through the reaction

\[ \text{Gd}_2\text{O}_3 + 3\text{H}_2\text{O} \rightarrow 2\text{Gd(OH)}_3 \]

Co oxidation under negative \( V_c \) occurs by the reverse process (Fig. 2b), with Pt acting as the anode for Co oxidation and Au acting as the cathode for \( \text{H}_2\text{O} \) recombination (see Supplementary Information section 4). In both the oxidation and reduction processes, it is expected that mass transport is dominated by protons instead of \( \text{O}^2^- \) owing to the much smaller size and lower activation energy for hopping of the former.47,48

If positive \( V_c \) pumps \( \text{H}^+ \) as proposed, sustained bias application should lead to hydrogen accumulation and evolution at the bottom electrode, and this is indeed observed. Figure 2c shows a scanning electron microscope image of a Pt(3 nm)/GdOₓ(100 nm)/Au(3 nm) film after applying \( V_c = +3 \text{ V} \) for 5 h. The inset optical micrograph shows bubble formation, which after cross-sectioning is revealed to arise from gas evolution and GdOₓ delamination at the bottom electrode, which could only be \( \text{H}_2 \) since \( \text{O}_2 \) evolution could only occur at the anode. Formation of \( \text{H}_2 \) bubbles under similar conditions has also been recently observed in Pt/TiOₓ/Pt memristors.49

Figure 2d shows the voltage threshold for the onset of PMA in a CoO structure to be about 1.5 V, which agrees well with the proposed reactions in equations (1) and (2).49 This threshold is independent of GdOₓ thickness \( t_{\text{GdO}_x} \) as seen in Fig. 2d, implying that the process is dictated by an electrochemical reaction rather than by an electric field. Cyclic voltammetry measurements (Supplementary Fig. 4f and g) provide further support for the proposed reaction chemistry.49

### Magnetic anisotropy control by proton injection

We now show that hydrogen insertion at the Co/GdOₓ interface allows the anisotropy to be toggled from out-of-plane to in-plane without requiring redox reactions in the ferromagnetic Co (Fig. 3a). Surface anisotropy is known to be sensitive to adsorbed \( \text{H} \), as shown previously for ultrathin ferromagnetic films in ultrahigh vacuum upon exposure to molecular or atomic hydrogen.46,47 Here, we show that the same behaviour can be gated in solid-state devices. Starting from a virgin state with PMA (Fig. 3b), the magnetization rotates in-plane when \( V_c = +3 \text{ V} \) is applied for 800 s (Fig. 3c), corresponding to accumulation of hydrogen at the Co/GdOₓ interface. When \( V_c \) is set to 0 V (grounded), PMA is spontaneously recovered (Fig. 3d) as the accumulated hydrogen forms \( \text{H}^+ \) and diffuses away from the bottom electrode. In-plane magnetization reorientation is confirmed by Fig. 3e–g, which shows that the PHE signal is absent when the film has PMA and is present when the AHE signal vanishes (Fig. 3e–g) under positive bias. Similar to the reduction of CoO described above, the accumulation of hydrogen that causes this magnetization reorientation transition only occurs in the presence of humidity, as shown in Supplementary Fig. 7.

Figure 3h–j shows cycling results for a device with \( t_{\text{GdO}_x} = 4 \text{ nm} \), in which switching is much faster. \( V_c \) was cycled beyond 2,000 times between +3 V and 0 V at 0.5 Hz, and out-of-plane hysteresis loops were acquired at 25-ms intervals using a polar magneto-optical Kerr effect (MOKE) polarimeter (see Methods). Figure 3h shows the ratio of the remanent \( (M_r) \) to saturation magnetization \( (M_s) \) as a function of time, for cycles 1–10 and 2,060–2,070, tracking the in-plane/out-of-plane transitions (Fig. 3i). The square out-of-plane loop in the virgin state (Fig. 3i) is indistinguishable from the loop after toggling the magnetization in plane, both after the first cycle (Fig. 3i) and after cycle 2,070 (Fig. 3j). We find, however, that the response time degrades slightly with repeated cycling (Fig. 3b), which may be associated with increased leakage currents in the oxide. The switching times at the rising and falling \( V_c \) edge are 100 ms and 400 ms respectively (Supplementary Fig. 10). This switching speed is faster than any room-temperature results in the literature for magneto-ionic switching.48 The asymmetry in switching can be mitigated by applying a negative \( V_c \) to accelerate \( \text{H}^+ \) removal from the interface, but this can also lead to Co oxidation, which leads to a progressive irreversible degradation of PMA owing to the irreversibility of oxygen insertion into the magnetic layer.49 (Supplementary Figure 9).

The electrochemical reactions at \( V_c = +3 \text{ V} \) in the two half cells (top and bottom electrodes), given by:

\[ \text{Anode (Au): } 2\text{H}_2\text{O} \rightarrow 4\text{H}^+ + \text{O}_2 + 4\text{e}^- \] (3)

\[ \text{Cathode (Pt): } 4\text{H}^+ + 4\text{e}^- \rightarrow 4\text{H} \] (4)
cannot proceed without electron flow through the external circuit. Thus, one can realize two operating modes by either grounding the device \( (V_c = 0 \text{ V}) \) or leaving the device at open circuit when \( V_c \) is removed. Figure 4a shows that after \( V_c = +3 \text{ V} \) is applied, the magnetization transitions from out-of-plane (Fig. 4b) to in-plane (Fig. 4c), and when \( V_c \) is set to open circuit, the in-plane state is retained (Fig. 4c and d). This implies that the accumulated hydrogen remains at the interface. In fact, for a device with very low leakage, this hydrogen-loaded state can be retained for more than three days under ambient conditions and at least a week under vacuum,
as shown in Supplementary Information section 5. When the device is subsequently set to closed circuit, PMA is spontaneously recovered (Fig. 4e). Hence, the magnetization state can be switched in a nonvolatile fashion between the out-of-plane and in-plane states, or toggled with a unipolar voltage, depending on whether the \( V_G = 0 \) condition is at open or closed circuit. This ability to modify the magnetic state over two very distinct timescales can be particularly attractive for neuromorphic computing where one can modify the chemical potential (Nernst potential) that arises in the hydrogen-loaded state. During this measurement, we simultaneously probed the time-dependent Co magnetic orientation (Supplementary Fig. 6).

These results show that removal of H from the bottom electrode requires the reactions in equations (3) and (4) to occur in reverse. To be removed, H must first split into \( \text{H}^+ \) and \( e^- \) at the bottom electrode, so that the \( \text{H}^+ \) can be transported back up through the GdO\(_x\) to the top electrode, where it recombines with atmospheric oxygen. If there is no electronic conduction path, the reaction cannot proceed and as a result the in-plane magnetized state is retained at open circuit. Hence, a finite leakage current through the GdO\(_x\) reduces the stability of the hydrogen-loaded state (Supplementary Information section 5) under ambient conditions, because it offers an alternate path for electron transfer from the bottom electrode to the top electrode. In Supplementary Information section 5, we also show that, when the device is in vacuum, equation (3) becomes rate-limiting, and the out-of-plane state cannot be recovered even when applying a reverse bias. From this, we conclude that H cannot simply diffuse from the bottom electrode; its insertion and removal are governed by the anodic and cathodic electrochemical reactions in equations (3) and (4).

Next, to quantify the amount of hydrogen needed to achieve the in-plane state, we also measured the transient (discharge) current flowing in the opposite direction when \( V_G \) is set to 0 V, driven by the chemical potential (Nernst potential) that arises in the hydrogen-loaded state. During this measurement, we simultaneously probed the time-dependent Co magnetic orientation (Supplementary Fig. 6). From the integrated charge, we found that approximately 1 H for every 10 Co atoms is needed to retain the in-plane switched state.

**Voltage control of a heavy-metal/ferromagnet interface**

Since Pd is well known for its hydrogen loading capacity\(^{25}\), and atmospheric hydrogen loading in Pd/Co/Pd has previously been shown to modulate PMA\(^{41}\), we next exploit this feature by inserting a Pd layer between Co and GdO\(_x\). Figure 5a shows the layer schematic of a Ta(4 nm)/Pd(3 nm)/Co(0.6 nm)/Pd(4.5 nm)/GdO\(_x\) (10 nm)/Au(3 nm) heterostructure. Because the Co layer is protected by Pd, negative \( V_G \) does not result in oxidation of Co (Fig. 5b), which allows for applying a negative gate bias to accelerate H removal and recovery of PMA. In this device, positive and negative \( V_G \) can pump hydrogen into and out of the Pd layer reversibly, switching
the anisotropy from out-of-plane to in-plane and back\(^{43}\) (Fig. 5c–e).

Figure 5f shows the switching cycles of \(M_r/M_I\) as \(V_C\) is cycled between +4 V and −1 V at 1 Hz. Robust switching was achieved, with a switching time of around 150 ms at both the rising and falling edges (Supplementary Fig. 11).

To provide direct evidence of insertion of H into the heavy-metal layer, we performed X-ray absorption spectroscopy (XAS) at the 23-ID-1 beamline at the National Synchrotron Light Source II (see Methods). Figure 5g shows an XAS spectrum of a virgin Ta(4 nm)/Pd(3 nm)/Co(≈0.6 nm)/Pd(4.5 nm)/GdO\(_2\) sample in the range between 525 eV and 575 eV, where one can observe the Pd M\(_3\) edge\(^{31}\) at about 532 eV and O K-edge at about 538 eV. The identity of the Pd M\(_3\) peak is verified by measuring the XAS spectrum with and without a Pd layer (Supplementary Fig. 12). Figure 5h shows comparison of the Pd M\(_3\) edge in a virgin and in a voltage-modified device. For the voltage-modified device, \(V_C = +3\) V is applied for >5 min ex situ before the XAS spectrum was measured.

From the data, we can clearly observe a peak shift of around +0.7 eV for the Pd M\(_3\) edge upon voltage application. This shift arises from two sources. First, previous XPS studies have shown that the binding energy of core electrons shifts slightly when Pd becomes PdH\(_x\). This shift is around +0.17 eV for the 3d electrons and its magnitude is very small because hydrogen in Pd is well screened in the lattice\(^{48}\). Second, there is a change in energy of the final unoccupied 4d states of Pd by around +0.7 eV when it becomes a hydride\(^{49,50}\). This change accounts for the largest part of the peak shift in the XAS spectrum. The total peak shift of the Pd M\(_3\) edge should therefore be around +0.87 eV, which is very close to the +0.7 eV peak shift we observe in our data. We have also confirmed this with thick Mg as the hydrogen loading layer\(^{48}\) and the results show that a substantial amount of hydrogen can be pumped into the system using this mechanism (Supplementary Fig. 13). Thus we have shown that the magnetic anisotropy at a metal/metal interface can be modulated substantially by an electric field through electrochemical gating of a metal to its hydride phase in an appropriately designed solid-state heterostructure. This effect cannot be achieved by any other known mechanism because electric fields vanish in a metal.

**Conclusions**

We have shown that hydrogen plays an essential part in previously observed magneto-ionic switching. We introduced a new switching scheme based on hydrogen accumulation and removal at a Co/GdO\(_2\) interface to achieve excellent cyclability and fast room-temperature operation. Depending on the application, the device can be operated in a volatile or non-volatile mode, allowing for device toggling or low-power state retention. The anisotropy changes that can be achieved correspond to magnetoelastic efficiencies of at least 5,000 \(\mu\)J V\(^{-1}\) m\(^{-1}\) (Supplementary Information section 14), similar to the maximum values reported for oxygen ion gating\(^{51}\), but without the associated change in chemical state. In terms of switching speed, this new mechanism compares favourably to other ion-mediated gating mechanisms. For instance, lithium intercalation\(^{52,53}\) takes hours to produce substantial magnetic changes in the device, and while this timescale can be reduced down to tens of minutes by increasing the charging current, this usually comes at the expense of cyclability. For oxygen–ion gating, ref. \(^{54}\) have demonstrated fast control of magnetism down to about 0.2 ms in a Co/SrCoO\(_{2.5}\) heterostructure at room temperature, but they were only able to tune the coercivity by a few oersteds, which is too little for practical devices. For oxygen–ion gating in Co/GdO\(_2\) stacks, typical response times are of the order of minutes to tens of minutes and the effect usually requires elevated temperatures\(^{54}\). Our achievement of 100 ms switching at room temperature, with robust behavior for >2,000 cycles, thus holds promise for applications such as neuromorphic computing, where switching rates of the order of 100 Hz can permit practical device implementations\(^{54}\). Although device retention at this stage is a few days, we expect substantial improvement with further engineering of the hydrogen storage material. We remark that the non-volatility of the gating effect is not a prerequisite for its utility, as many current proposed applications are based on dynamic modulation of magnetic anisotropy for efficient current-induced switching. Finally, we have also demonstrated voltage gating of a metal/metal interface by harnessing the hydrogen loading capability of noble metals such as Pd in a Pd/Co/Pd/GdO\(_2\) heterostructure. This paves the way towards solid-state voltage gating of other spin–orbital and exchange effects mediated by heavy metals with substantial hydrogen storage capacity, such as spin–orbit torques\(^{55}\), the Dzyaloshinskii–Moriya interaction\(^{57}\) and RKKY exchange coupling\(^{58}\) in buried ferromagnet/metal interfaces. Such devices can be implemented by using a symmetric heavy metal/ferromagnet/heavy metal structure, for instance, where we can generate inversion symmetry by loading hydrogen into one of the heavy-metal layers and in turn completely remove (or restore) the Dzyaloshinskii–Moriya interaction or spin–orbit torques using a gate voltage. Finally, to make this an entirely closed system, a

**Fig. 4 | Magnetic response under short circuit and open circuit.** (a) Evolution of out-of-plane magnetization remanence ratio \(M_r/M_I\) versus time and the corresponding gate voltage \(V_C\). The dashed line indicates open circuit while the solid line at \(V_C = 0\) V indicates short circuit. \(V_C = +3\) V is applied between time \(t = 30\) s and \(t = 100\) s, at which point the probe is lifted from the top electrode. At \(t = 5\), \(300\) s, the probe is landed again and \(V_C\) set to 0 V (ground).

(b)–(e) Polar MOKE hysteresis loops corresponding to \(t = 25\) s (b), \(t = 110\) s (c), and \(t = 4\), \(200\) s (d), and \(t = 5\), \(600\) s (e).
Fig. 5 | Voltage gating of metal/metal interface by exploiting hydrogen loading in Pd. a, Schematic of device operation in a Pd/Co/Pd/GdO3 cell. 

**Online content**

Any methods, additional references, Nature Research reporting summaries, source data, statements of data availability and associated accession codes are available at https://doi.org/10.1038/s41563-018-0211-5.

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**References**


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Author contributions

A.J.T. and G.S.D.B. conceived and designed the experiments. G.S.D.B. supervised the project. H.L.T. provided insight into the reaction processes and mechanisms. A.J.T. fabricated the samples with assistance from M.H. C.O.A. and A.J.T. conducted the anomalous and planar Hall measurements. F.B., W.H., C.M. and A.J.T. performed the XAS measurements. S.W. provided insights on the XAS data. A.J.T. performed the MOKE measurements with help from M.M. A.J.T. wrote the manuscript with guidance from G.S.D.B. and H.L.T. A.J.T. and G.S.D.B. conceived and designed the experiments. G.S.D.B. supervised the project. H.L.T. provided insight into the reaction processes and mechanisms. A.J.T. fabricated the samples with assistance from M.H. C.O.A. and A.J.T. conducted the anomalous and planar Hall measurements. F.B., W.H., C.M. and A.J.T. performed the XAS measurements. S.W. provided insights on the XAS data. A.J.T. performed the MOKE measurements with help from M.M. A.J.T. wrote the manuscript with guidance from H.L.T. and G.S.D.B. All authors discussed the results.

Competing interests

The author declares no competing interests.

Additional information

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Methods
Sample preparation. Ta(4 nm)/Pt(3 nm)/Co(0.9 nm)/GdO\textsubscript{x}(t\textsubscript{GdO\textsubscript{x}}(nm))/Au(3 nm) films were fabricated on thermally oxidized Si (100) substrates using magnetron sputtering at room temperature and 3 mTorr Ar pressure. The metal layers were grown by direct-current sputtering. The GdO\textsubscript{x} layer was deposited either using reactive sputtering with \(p_{\text{O}_2}\) of 0.07 mTorr or radio-frequency sputtering with \(p_{\text{O}_2}\) of 0.7 mTorr \text{O}_2. For the samples described in Fig. 1 with Co in the initially oxidized state, the Co layer was reactively sputtered with \(p_{\text{O}_2}\) of 0.07 mTorr \text{O}_2 with a deposition time corresponding to the time required to deposit 0.9 nm of metallic Co. For AHE and PHE measurements, the structure is patterned into a Hall cross geometry with 500 \(\mu\text{m}\) arm width and with Au(3 nm) deposited over the 0.25 mm\(^2\) active region to serve as a gate electrode. For MOKE measurements, 200- \(\mu\text{m}\) diameter Au(3 nm) electrodes were patterned on top of the GdO\textsubscript{x} layer of a continuous film, with the Ta(4 nm)/Pt(3 nm) underlayer uncovered by GdO\textsubscript{x} at the sample edge to allow electrical contact to the back. All patterning was done using shadow mask lithography. The measured leakage current in the GdO\textsubscript{x} films is <1 nA at \(V_G = +2\) V for devices with diameter 200 \(\mu\text{m}\) and 30-nm-thick GdO\textsubscript{x}.

Hall effect measurements in different atmospheres. Anomalous Hall effect (AHE) and planar Hall effect (PHE) measurements were performed using a lock-in amplifier with an alternating-current injected current of amplitude 2 mA and frequency 1 kHz. For the AHE measurements, the field was swept perpendicular to the plane; for the PHE measurements, the field was oriented in the sample plane, at 45° to the current flow axis. AHE and PHE hysteresis loops were acquired using a 2-s field sweep time. The measurements were performed in a modified CVX-FF Lakeshore probe station with a base vacuum pressure of about 10\(^{-9}\) mbar. Experiments under controlled gas environments were performed by backfilling the chamber with either \text{O}_2 gas (99.999% purity) or \text{N}_2 gas (99.999% purity). Humidity was introduced into the \text{N}_2 gas flow by bubbling through water. Wet \text{N}_2 and ambient conditions at 25 °C correspond to about 20 mtorr and 12 mtorr of \text{H}_2\text{O} partial pressure respectively. All experiments were performed at room temperature.

Polar MOKE measurements. MOKE measurements were performed using a 1-mW laser with a wavelength of 660 nm focused to a spot size of about 10 \(\mu\text{m}\). Experiments were performed in polar geometry and hence sensitive to the out-of-plane magnetization component. To apply gate voltage \(V_G\) to the circular electrodes, a CuBe probe was placed near the edge of the electrode and the Ta/Pt back electrode was grounded. The laser spot was focused on the middle of the electrode. All experiments were performed at room temperature.

XAS. XAS spectra were acquired at the Coherent Soft X-ray Scattering beamline at the National Synchrotron Light Source II, Brookhaven National Laboratory using fluorescent yield. The incident soft X-ray beam has a footprint of about 200 \(\mu\text{m}\) and the sample is tilted 15° relative to the incident beam. The sample used for the measurement has a Hall bar geometry with sample structure Ta(4 nm)/Pd(3 nm)/Co(0.6 nm)/Pd(4.5 nm)/GdO\textsubscript{x}(30 nm) and a 3 nm Au top gate. The main chamber base pressure is about 2 \(\times\) 10\(^{-9}\) mtorr, and the sample is kept at 100 K throughout the measurement.

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