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Direct observation of the perpendicular shape anisotropy and thermal stability of p-STT-MRAM nano-pillars

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Perpendicular shape anisotropy (PSA) offers a practical solution to downscale spin-transfer torque Magnetic Random-Access Memory (STT-MRAM) beyond the sub-20 nm technology node whilst retaining thermal stability of the storage layer magnetization. However, our understanding of the thermomagnetic behavior of PSA-STT-MRAM is often indirect, relying on magnetoresistance measurements and micromagnetic modelling. Here, the magnetism of a FeCoB / NiFe PSA-STT-MRAM nano-pillar is investigated using off-axis electron holography, providing spatially resolved magnetic information as a function of temperature, which has been previously inaccessible. Magnetic induction maps reveal the micromagnetic configuration of the NiFe storage layer (~ 60 nm high, \leq 20 nm diameter), confirming the PSA induced by its 3:1 aspect ratio. *In-situ* heating demonstrates that the PSA of the FeCoB / NiFe composite storage layer is maintained up to at least 250 °C, and direct quantitative measurements reveal the very moderate decrease of magnetic induction with temperature. Hence, this study shows explicitly that PSA provides significant stability in STT-MRAM applications that require reliable performance over a range of operating temperatures.

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1. Introduction

Magnetic random-access memory (MRAM) is a non-volatile memory based on the storage of one bit of information by a ferromagnetic memory cell. The discovery of the spin-transfer torque (STT) effect has made MRAM industrially relevant, thanks to the ability to write cells with an electric current. STT-MRAM can be easily integrated with CMOS technology⁶, has low energy consumption⁷, superior endurance^{7,8}, and rather high areal density⁹. In practice, STT-MRAM involves the use of a magnetic tunnel junction (MTJ) comprising an MgO tunnel barrier (1 – 1.5 nm thick) separating two thin perpendicularly-magnetized layers, one magnetically-pinned reference layer, and one switchable storage layer. This design is called p-STT-MRAM, the perpendicular anisotropy resulting from interfacial electronic hybridization effects occurring at the interface between the magnetic electrodes and the oxide tunnel barrier¹⁰. The electrical resistance of the MTJ changes significantly when the layers are magnetized in parallel and antiparallel states, providing a system of readable / writable ‘0’ or ‘1’ binary information. Increasing the areal bit density of p-STT-MRAM requires to reduce the in-plane size or diameter of the nano-patterned MTJ¹¹. However, below a characteristic magnetic length scale around 20nm, this comes with an excessive decrease of the thermal stability of the magnetic moment of the ultrathin storage layer. One solution is to increase the storage layer thickness to larger than its diameter, so that its out-of-plane aspect ratio combined with large volume, provides additional thermal stability through perpendicular shape anisotropy (PSA)^{12,13}. Previous studies have shown that the PSA-STT-MRAM are indeed highly thermally stable, making them a practical solution to downsize scalability of STT-MRAM at sub-20 nm technology nodes^{14,15}. However, our knowledge of the thermal stability of these STT-MRAM nano-pillars is often indirect, relying on magnetoresistance measurements and micromagnetic modelling. In order to understand fully their thermomagnetic behavior, it is necessary to examine the effect of temperature directly. The advanced transmission electron microscopy (TEM) technique of off-axis electron holography allows imaging of magnetization within nano-scale materials, with high spatial resolution and sensitivity to induction field components transverse to the electron beam¹⁶. Combining electron holography with *in-situ* heating within the TEM has already allowed direct imaging of the thermal stability of nano-scale signal carriers and fields of magnetic minerals¹⁷⁻¹⁹, meteorites²⁰ and pre-patterned MTJ conducting pillars²¹.

In this paper, we use electron holography to image the micromagnetic configuration of an individual ≤ 20 nm diameter FeCoB / NiFe STT-MRAM nano-pillar and acquire quantitative measurements of magnetic induction from the high-aspect-ratio FeCoB / NiFe composite free

layer. In addition, we experimentally demonstrate the influence of PSA on its thermal stability through *in-situ* heating within the TEM to 250 °C. This study provides direct evidence of the PSA exhibited by the high-aspect-ratio free layer, and its improved thermal stability compared to standard p-STT-MRAM.

2. Experimental

An array of FeCoB / NiFe PSA-STT-MRAM nano-pillars was fabricated through sequential e-beam lithography, reactive ion etching and ion beam etching¹². The full stack of PSA MTJs has the following composition: SiO₂ / Pt (25 nm) / SAF / Ta (0.3 nm) / FeCoB (1.1 nm) / MgO (1.2 nm) / FeCoB (1.4 nm) / W (0.2 nm) / NiFe (60 nm) / Ta (1 nm) / Ru (3 nm) / Ta (150 nm), where the thick NiFe layer provides the PSA^{12,14}. SAF stands for a synthetic antiferromagnet, providing a compensated-moment antiferromagnetically-pinned magnetic reference as the bottom layer. A protective layer of organic resin (~ 1 μm) was spin-coated onto the array prior to being inserted into a Thermo Fisher Strata 400S dual-beam focused ion beam (FIB) / secondary electron microscope (SEM) for TEM sample preparation. A protective W layer was deposited over the resin and trenches were FIB irradiated until a ~ 3 μm × ~ 20 μm lamella was prepared and transferred to Omniprobe copper lift-out grids, where it was thinned to a thickness of ~ 300 nm using conventional FIB methods. The protective resist layer was etched away using a plasma cleaner and the remaining W layer was broken off with the micromanipulator. High angle annular dark field (HAADF) scanning TEM (STEM) imaging was performed using a probe-C_s-corrected Thermo Fisher Titan TEM at 200kV. Corresponding chemical analysis was provided by energy dispersive X-ray (EDX) spectroscopy using Bruker SDD Super X Fast EDX four-detectors. Off-axis electron holograms were acquired under field-free conditions in Lorentz mode on a Gatan OneView 4K camera using a Thermo Fisher Titan TEM equipped with an image-C_s corrector and an electron biprism. A voltage of 220 V was applied to the biprism, resulting in an interference fringe spacing of ~ 1.7 nm. Stacks of 8 electron holograms (each acquired for 4s) were aligned and then averaged using the Holoview software²² to improve the signal to noise ratio of the reconstructed phase images. The magnetization states of the nano-pillars were visualized through separation of the magnetic contribution to the phase shift from the mean inner potential (MIP), achieved by tilting to ± 40° and applying the strong field of the objective lens (< 1.5 T) to reverse the magnetic contribution²³. *In-situ* heating up to 250°C was performed using a Gatan heating holder under field-free magnetic conditions, allowing 30 minutes to stabilize from thermal drift at each temperature interval. The heating

was repeated and the magnetic reversal was performed at each temperature interval to isolate the MIP, and subtracted from the first heating to reconstruct the thermomagnetic behavior of the nano-pillars^{18,19}. For the construction of magnetic induction maps, the cosine of the magnetic contribution to the phase shift was amplified ($\times 150$) to produce magnetic phase contours and arrows were added to show the direction of the projected induction.

3. Results

Figure 1 presents the chemical composition, morphology and structure of an individual nano-pillar from the etched FeCoB / NiFe stack. The HAADF STEM image (Fig. 1a) displays the general morphology of the nano-pillar with a distinct difference in Z-contrast between the top Ta mask and lower shaft. This is confirmed by EDX chemical mapping of Fig. 1b, revealing the NiFe section of the nano-pillar is ~ 60 nm high with a diameter of ≤ 20 nm, and is separated from the hard Ta mask by the ~ 3 nm Ru layer. The O signal is dispersed across the entire nano-pillar and its physical state is explored through precession diffraction acquired from the boxed region (red) in the EDX chemical map of Fig. 1c. The weakly crystalline / amorphous state of the surface of the nano-pillar highlighted in the scanned area of Fig. 1d (left) is demonstrated in the associated electron diffraction image (right). Conversely, Fig. 1e shows the crystalline state of the center of the nano-pillar in the scanned area (left) and associated electron diffraction (right). The line profile of Fig. 1f is acquired from the red arrow in Fig. 1c (averaged vertically over ~ 15 nm) and displays the cross-section of normalized x-rays % for Fe, Ni and O content. The Ni and Fe profiles overlap consistently and the diffraction data of Fig. 1e demonstrates a homogeneous, crystalline NiFe core of the nano-pillar with measured composition of 81 at% Ni and 19 at% Fe. The O-rich phase is confined to the surface and considering its weakly crystalline / amorphous state, is largely attributed to residual organic resin from the preparation process.

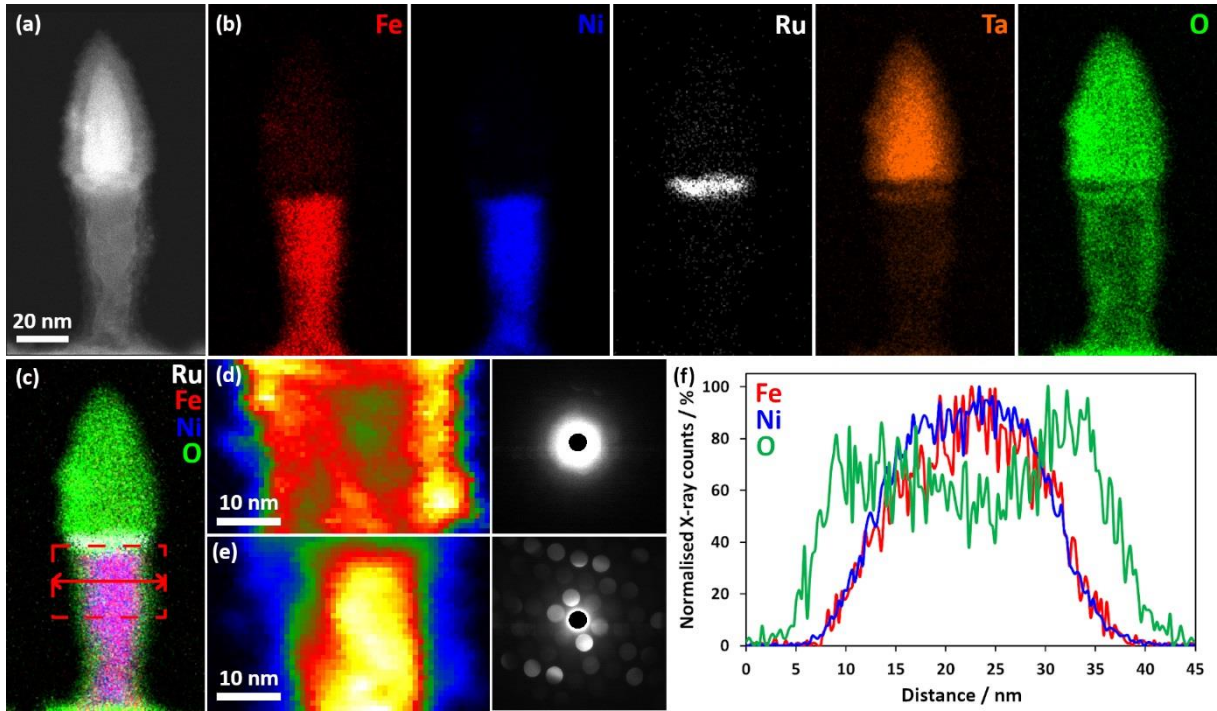


Figure 1. Overview of the morphology, chemical composition and structural properties of the nano-pillar. (a) HAADF STEM image of the nano-pillar; and (b) associated EDX chemical maps showing the elemental distribution of iron, nickel, ruthenium, tantalum and oxygen. (c) EDX chemical map highlighting the iron / nickel-rich center and oxygen-rich surface of the nano-pillar. (d,e) Precession electron diffraction acquired from the boxed region (red) in (c), showing the (d) amorphous sidewall surface; and (e) crystalline core. (f) Elemental line profile acquired along the red arrow in (c) and averaged vertically over ~ 15 nm, displaying the cross-section of normalized x-rays % for iron, nickel and oxygen content.

Figure 2 presents phase information reconstructed from electron holography of the nano-pillar, including quantification of magnetic induction and associated magnetization. As a guide, the MIP contribution (Fig. 2a) shows the morphology of the nano-pillar at the same magnification as the associated projected magnetic induction map (Fig. 2b), which clearly demonstrates the existence of a magnetic dipole within the nano-pillar. This is emphasized by superimposing the associated magnetic induction over the EDX chemical map (Fig. 2c), where the magnetic contours flow along the major axis of the NiFe section. A line profile of the magnetic phase contribution (ϕ_m) acquired along the white arrow in Fig. 2c (averaged vertically over ~ 15 nm) is overlaid over the chemical profile from the same region (Fig. 2d). The magnetic phase shift $\Delta\phi_m$ in the pillar can be used to quantify the integrated in-plane magnetic induction related to the nano-pillar, where the minimum and maximum ϕ_m values [Fig. 2c, dotted vertical lines (pink)] coincide with the full width half maximum of the Ni / Fe chemical profiles. Extracting quantitative calculations of magnetization from such local observations must be handled with care as it displays integrated information (see Supplementary materials for details on the calculations summarized below). At first approximation, the nano-pillar can be treated as a

uniformly-magnetized infinite cylinder of radius r . The magnetic induction transverse to the electron beam direction is considered to arise from magnetization only (neglecting dipolar fields, i.e., stray field and internal demagnetizing field *i.e.*) and can be estimated with the Aharonov-Bohm effect using a radius of ~ 8.6 nm (obtained from Fig. 2d). A first value of $\mu_0 M_s = 0.82 \pm 0.06$ T is extracted with the standard deviation determined from the noise of free space. To better quantify this important parameter, we can estimate the associated demagnetizing field of such a structure with the F_{ijk} functions from Hubert's formalism using a prism approach²⁴, at the exact same location of the measurement of the integrated magnetic flux obtained with the phase slope. We then obtain a better estimation of $\mu_0 M_s = 1.19 \pm 0.08$ T with the removal of the integrated demagnetizing and stray field globally antiparallel to magnetization, which lowered our first approximation. Subsequently, to overcome the geometrical approximation for the integrated demagnetizing field estimation with a prismatic geometry, we performed a tomographic reconstruction through inverse Abel transformation²⁵ to extract the magnetic flux density in the core vicinity of the cylinder. The resulting value was finally adjusted with the local estimation of demagnetizing field through F_{ijk} functions, as the tomography method helps to deconvolve the B field but not the internal H_d . Our final extraction leads to a value for the spontaneous magnetization induction of the NiFe nano-pillar of **$\mu_0 M_s = 1.14 \pm 0.01$ T**. This value is in reasonable agreement with the spontaneous induction for a $\text{Ni}_{81}\text{Fe}_{19}$ chemical composition, expected to reach 1.05 T²⁶. We ascribe the difference to uncertainties related to the exact value of the composition of the alloy the diameter of the PSA, and the sharpness of its outer surface. Being systematic, these errors are expected to have no impact on the thermomagnetic measurements reported in the following. More details in our analysis of this quantification are given in the supplementary materials.

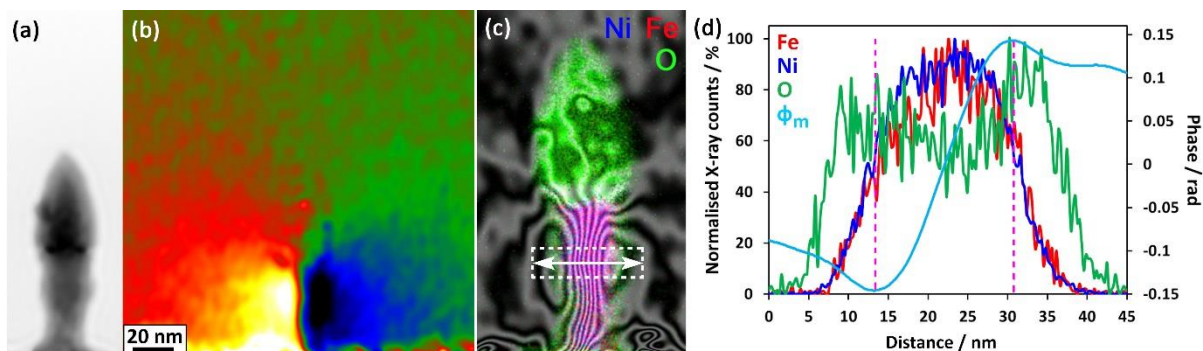


Figure 2. Overview of the magnetic induction associated with the nano-pillar and its quantification. (a,b) Reconstructed (a) MIP; and (b) magnetic contribution to the total phase, revealing the PSA along the major axis of the nano-pillar. (c) Combined EDX / magnetic induction map showing magnetic contours flowing along the major axis of the NiFe section. The contour spacing is 0.042 rad (cosine amplified by 150 times). (d) Line profile acquired along the white arrow in (c) and averaged vertically over ~ 15 nm around the mid-height of the PSA free layer, superimposing the cross-section of magnetic phase over the chemical profile.

Figure 3 presents the thermomagnetic behavior of the nano-pillar during *in-situ* heating from room temperature to 250 °C. The initial combined EDX / magnetic induction map of Fig. 3a displays the nano-pillar at 20 °C, with the magnetization pointing from the bottom to the top of the NiFe free layer and confirming its PSA. The associated magnetic induction maps of Figs 3b-h display the same nano-pillar during heating from 100 °C to 250 °C at 25 °C intervals. It is evident that the NiFe section retains its upward direction of magnetization at all temperatures up to 250 °C. The thermomagnetic stability of the NiFe section in the nano-pillar is supported by Figure 4, which presents corresponding quantitative measurements of magnetic induction at each temperature interval. The $\Delta\phi_m$ was measured from the same area presented in Fig. 2c,d and \vec{B} calculated using equation S2 (Supplementary information), which is proportional to magnetization. The average value of \vec{B} is $\sim 0.8 \pm 0.06$ T and the trend-line exhibits a decrease of ~ 0.08 T between 20 °C and 250 °C, which is in good agreement with the thermal behavior of bulk permalloy ($\text{Ni}_{80}\text{Fe}_{20}$)^{27,28}. As the contribution of dipolar field to the measured induction field is proportional to magnetization for a PSA, we expected that Fig.4 is representative of the thermal decay of magnetization in the PSA.

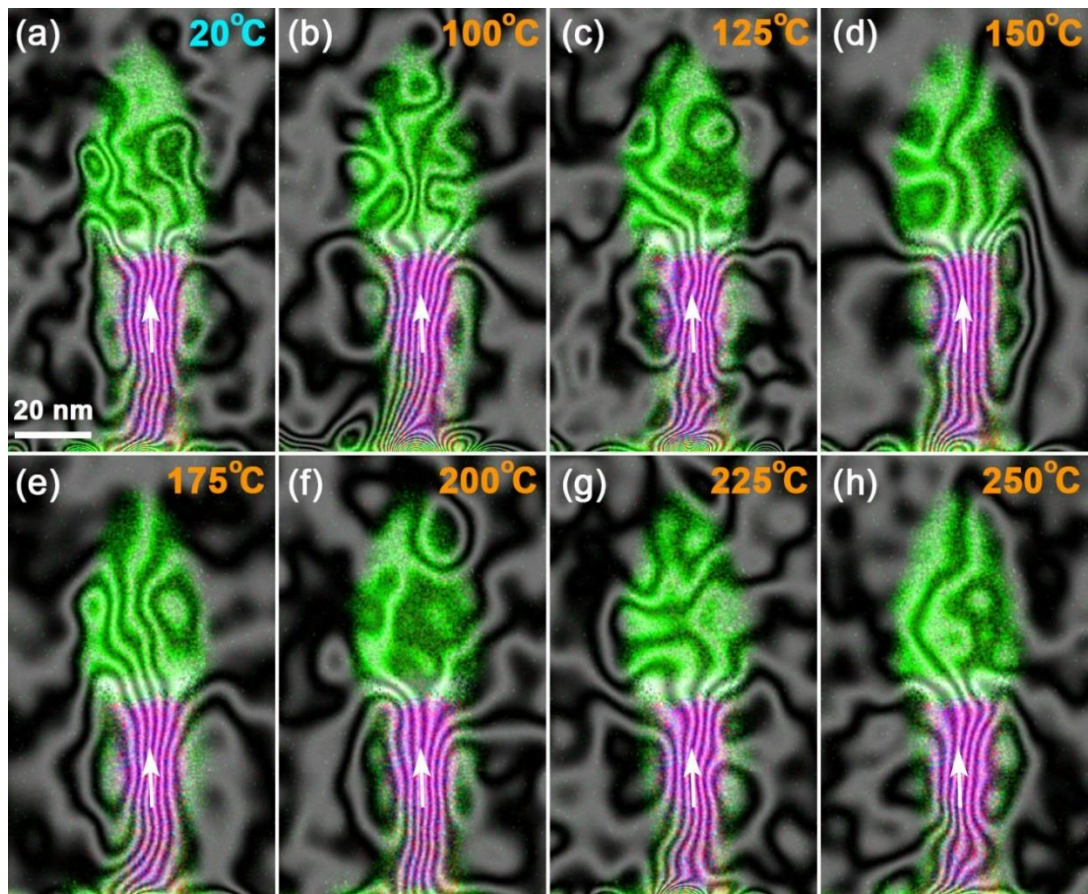


Figure 3. Visualization of the thermomagnetic stability within the nano-pillar. (a-h) Combined EDX / magnetic induction maps showing the PSA in the NiFe section of the nano-pillar acquired at (a) 20 °C; or during *in-situ* heating at 25 °C intervals from (b) 100 °C to (h) 250 °C. The contour spacing is 0.042 rad for all the magnetic induction maps (cosine multiplied by 150 times) and the magnetization direction is shown using arrows.

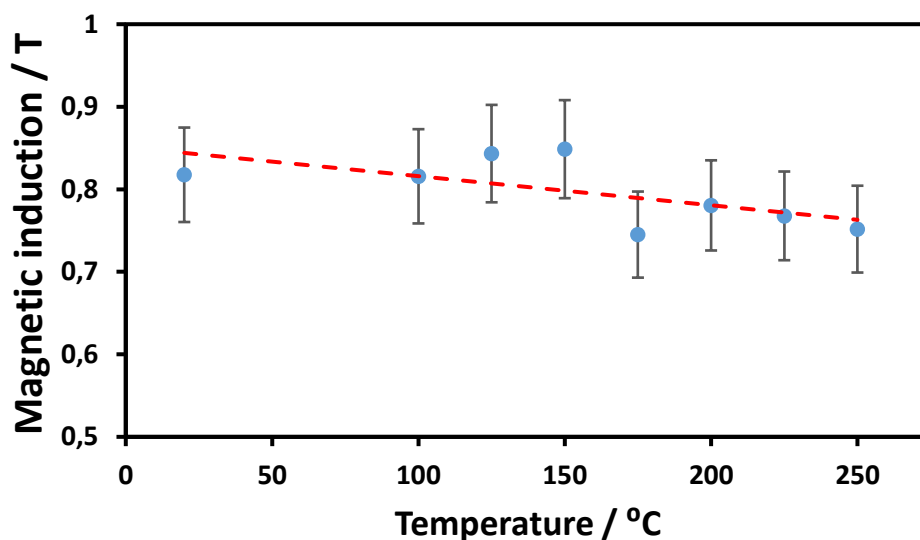


Figure 4. Magnetic induction associated to the nano-pillar as a function of temperature. Calculated magnetic induction transverse to the electron beam using the direct measurement of magnetic contribution to the phase from the NiFe section shown in Figure 3 with temperature, i.e., including a contribution from the demagnetizing and stray fields globally opposite to magnetization in the pillar, from 20 °C to 250 °C.

4. Discussion

The main reason to fabricate STT-MRAM nano-pillars with PSA is to stabilize and preserve the magnetic configuration of the free FeCoB / NiFe composite layer in response to temperature fluctuations during operation. It is evident from the *in-situ* heating TEM experiment that the dipolar magnetic orientation along the major axis of the NiFe section (bottom to top) is retained at all temperature intervals up to at least 250 °C. In addition, the associated direct measurements of \vec{B} (average $\approx 0.8\text{T} \pm 0.06\text{T}$) did not vary sufficiently to suggest a change in magnetic orientation, nor magnetization. The slight decreasing trend in magnitude of \vec{B} is attributed to an expected reduction in spontaneous magnetization when heating towards the Curie temperature²⁹, as would be observed in the same bulk material. As the shape-anisotropy energy barrier of the nano-pillar scales linearly with M_s^2 , this confirms that the energy barrier of switching between parallel and anti-parallel states has not been exceeded up to at least 250 °C, and the PSA has provided significant thermal stability compared to standard p-STT-MRAM stacks based on ultrathin films¹²⁻¹⁵. In standard p-STT-MRAM, the spontaneous magnetization may indeed decrease by a few tens of percent at 250°C due to enhanced fluctuations in low-dimensions for the ultrathin films involved, and the energy barrier associated with perpendicular anisotropy may decrease even more as it relies on interfacial magnetic anisotropy, which suffers from enhanced thermal decay due to its two-dimensional nature³⁰⁻³². Further, the maximum temperature of *in-situ* heating used (250 °C) far exceeds the operating temperatures for a range of STT-MRAM device applications, including commercial (70 °C), industrial (85 °C), automotive (125 °C) and military (150 °C)³³. It is also comparable to the temperature of reflow soldering used to attach chips to a printed circuit board (260 °C)³⁴. This thermal stability is crucial for many FLASH-replacement applications in which the program code is loaded in the chip prior to reflow soldering.

In conclusion, this electron holography study has provided explicit evidence of the PSA in ≤ 20 nm diameter p-STT-MRAM nano-pillar; previously only measured indirectly by magnetoresistance measurements and micromagnetic modelling. The enhanced thermal stability of PSA-STT-MRAM, compared with standard p-STT MRAM based on these films, has been confirmed by both imaging and quantitative measurements from their magnetic configuration during *in-situ* heating. This supports the use of PSA-STT-MRAM in a variety of applications that require reliable performance over a range of operating temperatures.

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