

High thermostability in CoFeB/MgO/Ta multilayers by $\{Pt(t)/Ta(t)\}_n$ superlattice buffer layers

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ABSTRACT

Ta/CoFeB/MgO/Ta multilayer known as the fundamental building block for magnetic tunneling junctions, has been studied for decades. However, its thermostability is normally less than 300 °C, which cannot meet the requirements for subsequent semiconductor processes. In this study, buffer layers of $\{Pt(t)/Ta(t)\}_n$ superlattices were introduced into CoFeB/MgO/Ta multilayers grown by magnetron sputtering. The growth period n and the single layer thickness t in the superlattice have been systematically explored. And it is found that the sample can maintain distinct perpendicular magnetic anisotropy (PMA) even after a 440 °C post-annealing treatment at $n = 3$ and $t = 1.5$ nm. This has been attributed to that the superlattice buffer layer might serve as a diffusion barrier, reducing Ta atoms from diffusing into the CoFeB/MgO interface. Less Ta at the interface results more FeO_x formed and enhances the hybridization of Fe 2p and O 3d orbitals which increases the thermostability of PMA. This suggests that superlattice buffer layer provides a new approach to improve the materials' thermostability.

1. Introduction

Perpendicular magnetic anisotropy (PMA) is one of the most critical properties of the magnetic layers used in magnetic random-access memories (MRAM), spin transistors, etc. [1–4] Magnetic tunnel junctions (MTJs) based on CoFeB/MgO with PMA are able to achieve higher speed, higher density, and lesser power consumption compared to the CoFeB/MgO with IMA [5]. However, CoFeB/MgO/Ta films, the most commonly seen material system used in MRAM and logic devices, usually transfer from PMA to in-plane magnetic anisotropic (IMA) when being annealed at above 300 °C [6,7]. On the other hand, a standard Complementary Metal Oxide Semiconductor (CMOS) process would generally require a thermal budget of 400 °C. The low thermostability of the CoFeB/MgO/Ta films seriously hinders their future applications with CMOS devices [8,9].

Therefore, many researches have been done to improve the thermostability of CoFeB/MgO/Ta films, such as searching for new substitute materials, optimizing process conditions, designing new stack structures, etc. [7,10–14] Among them, inserting buffer layers has long

been considered as a possible solution. Han et al. found that the maximum annealing temperature for samples can be increased from 270 °C to 300 °C by adding Pt underlayers [14]. Li et al. reported that by inserting a Hf layer between Ta and CoFeB, the multilayers could stand an annealing temperature as high as 350 °C [13]. Further research is still needed to improve the thermal stability to meet the requirements of a standard CMOS process.

In this study, superlattice buffer layers of $\{Pt(t)/Ta(t)\}_n$ have been introduced underneath CoFeB(1)/MgO(2)/Ta(3) (thickness in nm). All annealing treatments are carried out in vacuum. With the optimized parameters of $t = 1.5$ nm and $n = 3$, the film can maintain good PMA even after being annealed at 440 °C for 30 min. The multilayers were characterized using high-resolution transmission electron microscopy (HRTEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray photoelectron spectroscopy (XPS). The polycrystalline phase of the superlattice structure provides a uniform interface in favor of PMA. After annealing, the superlattice structure reduces the diffusion of Ta atoms to the CoFeB/MgO interface, increases the formation of FeO_x at the interface, and enhances orbital hybridization between Fe and O, resulting the

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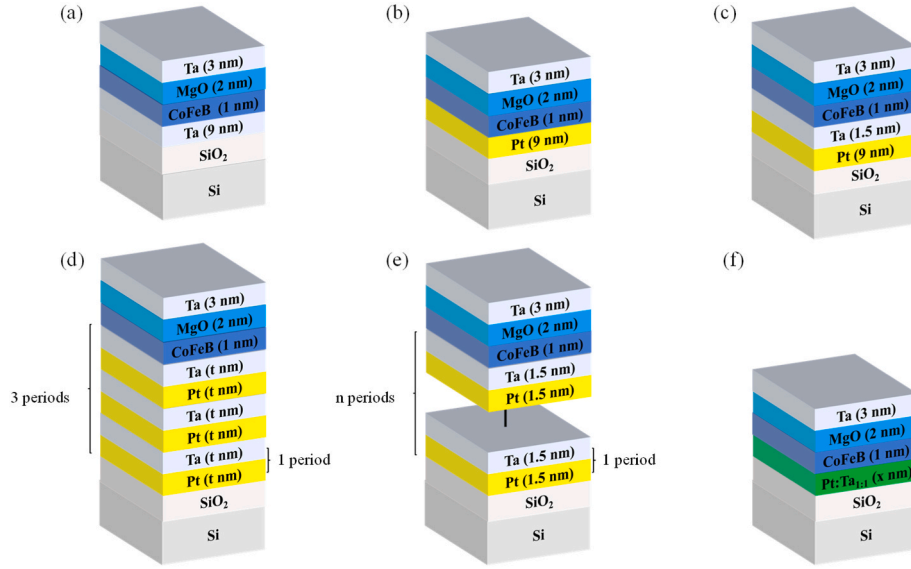


Fig. 1. The cross-sectional view of all the sample structures.

high thermostability of PMA up to 440 °C.

2. Experimental procedures

{Ta/Pt}_n/CoFeB/MgO/Ta multilayers were deposited on Si/SiO₂ substrates by magnetron sputtering with a base pressure of 3×10^{-7} Torr. The size of the substrate is 1×1 cm², and the diameters of all the target are 50.8 mm. The purity of Ta, CoFeB (4:4:2) and MgO (1:1) target is 99.95 %, while Pt is 99.99 %. The sputtering gas used is 99.999 % Ar for all the materials. For metal deposition, the pressure is kept at 6 mTorr and the DC power is kept at 10 W. For MgO deposition, the pressure is 10 mTorr and the RF power is 50 W. The deposition rates are 0.16 Å/s, 0.2 Å/s, 0.12 Å/s, 0.07 Å/s for Ta, Pt, CoFeB and MgO layers, respectively. During deposition, the substrates were kept at room temperature.

After the deposition, samples were annealed at different temperatures for 30 min in the sputtering chamber. The $M-H$ loops were measured at room temperature using the VSM of the models-EV7. The microstructure of the films was examined using HRTEM (Tallos F200X) and energy-dispersive X-ray spectroscopy (EDS). Chemical states on the

surface and at CoFeB/MgO interfaces were studied using XPS by ESCALAB 250 Xi from Thermo Scientific. The working vacuum is 5×10^{-8} Pa, and the X-ray source used is Al K α line with the photon energy of 1253.6 eV. The energy analyzer used is CAE with a pass energy of 30 eV. The depth information is realized by an in-situ ion beam etching, with an etching rate of 0.05 nm/s. During etching, the Ar pressure is 2×10^{-5} Pa with a beam energy of 2 KeV and a beam current density of 1 μ A/mm². All the XPS data are analyzed by Advantage. More detailed characterization information can be found in the supporting materials section, specifically in the Sample Characterization subsection.

3. Results and discussion

Samples with different structures were prepared, and their structures are as follows:

Sample A: Ta(9)/CoFeB(1)/MgO(2)/Ta(3)

Sample B: Pt(9)/CoFeB(1)/MgO(2)/Ta(3)

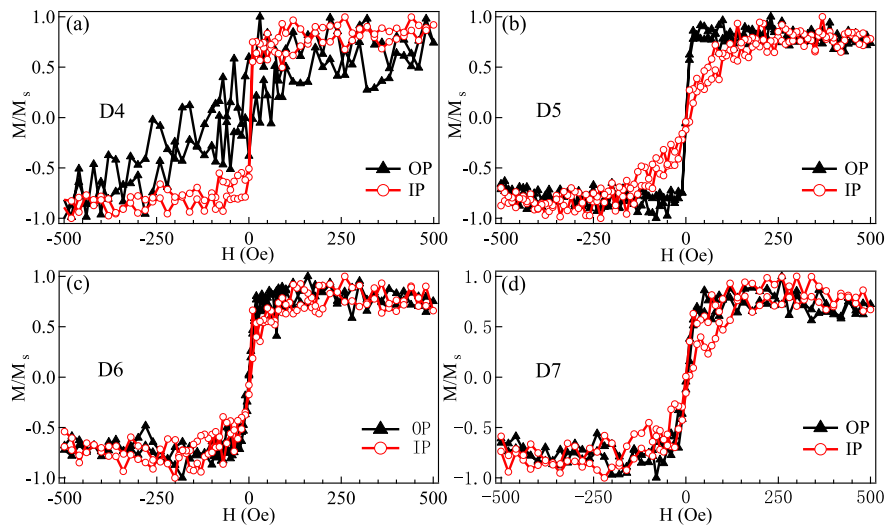


Fig. 2. Normalized hysteresis loops for samples D4-D7 after being annealed at 400 °C for 30 min. The sample D5 ($t = 1.5$ nm) demonstrates an out-of-plane easy axis, while all the others show IMA.

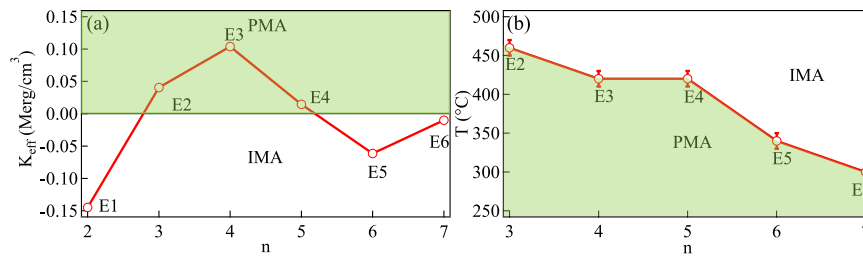


Fig. 3. (a) The K_{eff} of sample E series annealed at 400 °C. The sample E2-E4 have positive K_{eff} , suggesting PMA. (b) The critical annealing temperatures at which samples transition from PMA to IMA. Sample E1 exhibits IMA at all annealing temperatures.

Sample C: Pt(9)/Ta(1.5)/CoFeB(1)/MgO(2)/Ta(3)

Sample D series: $\{\text{Pt}(t)/\text{Ta}(t)\}_3/\text{CoFeB}(1)/\text{MgO}(2)/\text{Ta}(3)$

Sample E series: $\{\text{Pt}(1.5)/\text{Ta}(1.5)\}_n/\text{CoFeB}(1)/\text{MgO}(2)/\text{Ta}(3)$

Sample F series: $(\text{Pt}:\text{Ta}_{1:1})_x/\text{CoFeB}(1)/\text{MgO}(2)/\text{Ta}(3)$

The schematic diagrams of the samples are shown in Fig. 1.

Sample D series	D1	D2	D3	D4	D5	D6	D7	D8
t (nm)	0.3	0.6	0.9	1.2	1.5	1.8	2.1	2.4
Sample E series	E1	E2	E3	E4	E5	E6		
n	2	3	4	5	6	7		
Sample F series	F1	F2	F3	F4	F5	F6		
x (nm)	5	7	9	12	15	18		

Samples A, B, and C were designed as control groups, with different buffer layers of Ta(9), Pt(9) and Pt(9)/Ta(1.5), respectively. Samples A and C have out-of-plane easy axes when the annealing temperature is 280 °C and below, while they demonstrate in-plane anisotropy when annealed at 320 °C. On the other hand, sample B always show in-plane anisotropy when annealed at any temperature up to 320 °C (Fig. S1). This suggests Ta is better than Pt as a buffer layer, because the orbital magnetic moment of CoFeB grown on Ta with a bcc structure is higher than that grown-on Pt with a fcc structure [15], and this favors the formation of PMA.

Fig. 2 exhibits the normalized $M-H$ curves of samples D4 to D7, and all the samples were annealed at 400 °C. The black triangles and the red circles represent the hysteresis loops with the magnetic fields applied perpendicular or parallel to the film surface, respectively. Only sample D5 with $t = 1.5$ nm demonstrates an out-of-plane easy axis, while all the others show IMA. Fig. S2 shows samples D1-D3 and D8 have in-plane easy axes when the annealing temperature is 320 °C for 30 min.

To investigate the influence of the period numbers n on multilayer samples' thermostability, sample E series were prepared by varying the period number n from 2 to 7. Fig. 3a demonstrates the effective magnetic anisotropy energy (K_{eff}) of sample E series annealed at 400 °C, which are extracted from the $M-H$ curves as shown in Fig. S4. K_{eff} can be written as: $K_{\text{eff}} = H_k \times M_s/2$, where, H_k is the effective PMA field, and M_s is the saturation magnetization [16,17]. A negative K_{eff} value represents the IMA, while a positive K_{eff} value represents the PMA [18]. Samples E2 to E4 demonstrate PMA after annealed at 400 °C as shown in Fig. 3a. Furthermore, the critical annealing temperatures at which samples transition from PMA to IMA are shown in Fig. 3b. It peaks when n is 3, then gradually decreases with n . We have prepared F series, with co-sputtered Pt and Ta, as control samples. The in-plane behavior of the F series annealed at 280 °C and 320 °C. As shown in Fig. S5, the samples annealed at 320 °C displayed IMA in their MOKE curves. Above all, it can be concluded that the period number also plays a vital role in enhancing CoFeB/MgO/Ta multilayers' thermostability.

In order to investigate the origin of the enhancement on the thermostability, microstructures of the samples were characterized by

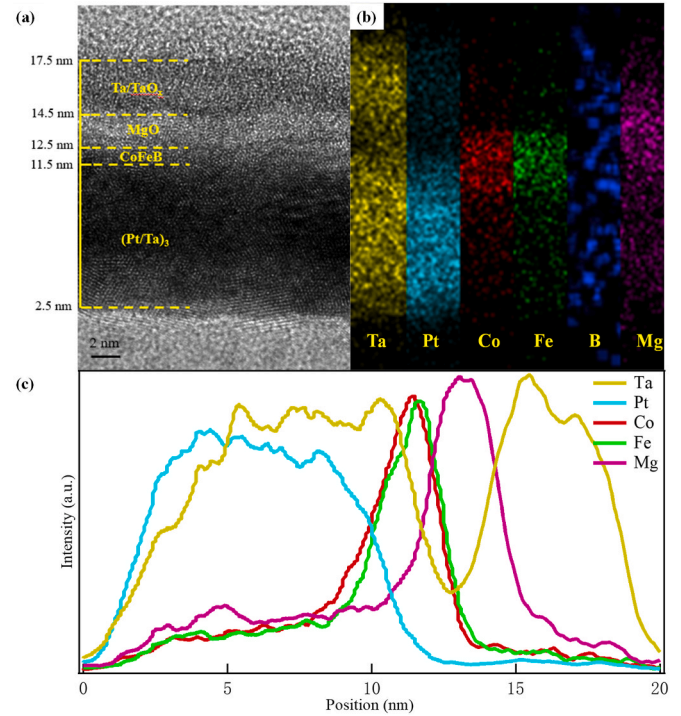


Fig. 4. (a) The cross-sectional TEM image of sample D5 after being annealed at 400 °C for 30 min. Each layer of the whole structure can be resolved, except $\{\text{Pt}/\text{Ta}\}_3$ superlattice. (b) EDX mapping images of Ta, Pt, Co, Fe and Mg, respectively. (c) Normalized intensity line profiles of b. A significant portion of Ta atoms have been diffused into CoFeB and MgO layers.

HRTEM and EDS. Fig. 4a shows a cross-sectional view of the HRTEM image for sample D5 ($\{\text{Pt}(1.5)/\text{Ta}(1.5)\}_3/\text{CoFeB}(1)/\text{MgO}(2)/\text{Ta}(3)$) annealed at 400 °C for 30 min. Every layer of the whole structure can be resolved with clearly interfaces, except $\{\text{Pt}/\text{Ta}\}_3$ superlattice buffer layer. There is no obvious interface between Pt and Ta layers, suggesting that the interdiffusion exists. However, the underlying buffer layers are crystallized and exhibits good uniformity as well as flatness, which are crucial for high quality growth of CoFeB layer [19].

Fig. 4b demonstrates the EDX mapping images of different elements, which show a reasonably good distribution within their corresponding layers. However, clearly interlayer diffusion exists for all of them, probably due to the high temperature annealing. Fig. 4c shows the intensity line profiles of the EDX maps, from which Ta, Pt, CoFeB, and MgO layers can be easily distinguished from each other. According to Fig. 4c, Pt and Ta shows flat plateau within the superlattice region, with a small fluctuation, which coincides with the HRTEM analysis and validates the occurrence of interdiffusion between Pt and Ta layers. Polycrystalline buffer layers with smoother surfaces might produce a more stable PMA under high-temperature annealing [19]. It is important to

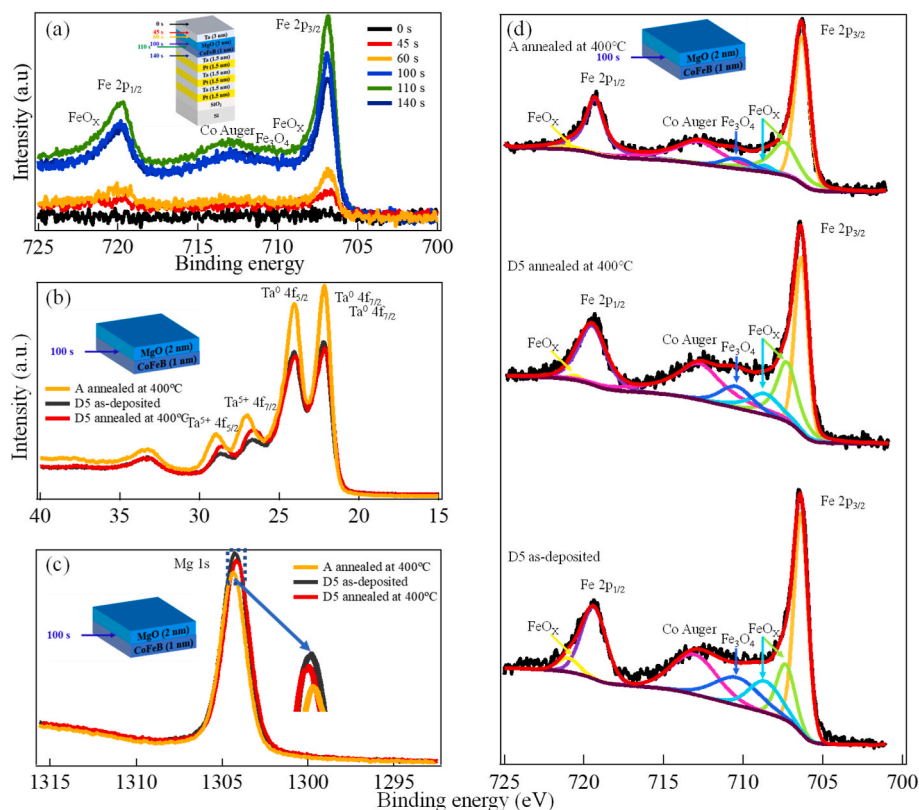


Fig. 5. (a) XPS spectra of Fe 2p for sample D5 annealed at 400 °C after Ar⁺ etching for different times. The arrows in the inset mark the probe depth of the XPS spectra. (b) Ta 4f XPS spectra for sample A annealed at 400 °C, sample D5 as-deposited and annealed at 400 °C, respectively. All of them have clearly Ta⁵⁺ peaks suggesting the formation of TaO_x at the CoFeB/MgO interface. (c) XPS spectra of Mg 1s. The peak heights demonstrate that there is the lesser Mg²⁺ in the annealed samples due to the decomposition of MgO at high temperatures. (d) Fe 2p XPS spectra at the CoFeB/MgO interface.

Table 1

Shows the Fe fitting parameters and the intensity of Ta, TaO_x, Mg1s for the films.

Sample	A annealed at 400 °C	D5 as-deposited	D5 annealed at 400 °C
Ta (Ta ⁰ 4f _{7/2})	8.22×10^4	6.03×10^4	5.79×10^4
TaO _x (Ta ⁵⁺ 4f _{7/2})	3.19×10^4	2.25×10^4	2.63×10^4
Mg 1s	2.43×10^5	2.61×10^5	2.54×10^5
S _{FeO_x} / S _{Fe+FeO_x}	32 %	41 %	43 %
Magnetic anisotropy	IMA	IMA	PMA

notice that a significant portion of Ta atoms have diffused into the CoFeB/MgO interface, which will also be seen by XPS analysis in the following.

To understand the microscopic mechanisms of the improved thermostability, depth-resolved XPS analysis has been performed as shown in Fig. 5 [20]. An Ar⁺ etching method was applied to remove the upper layers to obtain the XPS spectrum at different depth. The etching rate was selected to be 0.5 Å/s and the etching times were 0 s, 45 s, 60 s, 100 s, 110 s and 140 s. So that the XPS probes the chemical states at the surface, Ta layer, Ta/MgO interface, the MgO/CoFeB interface, CoFeB layer and Ta layer, respectively, as indicated by the arrows in the inset of Fig. 5a.

Fig. 5a shows XPS spectra of Fe 2p for sample D5 annealed at 400 °C. There are three peaks corresponding to metallic Fe 2p_{3/2}, Fe 2p_{3/2} in FeO_x, Fe₃O₄, the Auger peak of Co, metallic Fe 2p_{1/2}, Fe 2p_{1/2} in FeO_x, respectively. As the etching time increases, the peak of Fe also increases, reaching its maximum at 110 s, indicating that the CoFeB layer was exposed at 110 s. 100 s corresponds to the CoFeB/MgO interface. The FeO_x peaks in the blue line suggests that Fe at the CoFeB/MgO interface

is partially oxidized, which is also consistent with many other studies. [7,21].

Fig. 5b demonstrates the XPS spectra of Ta 4f for sample A and sample D5 w/o anneal. All the samples were etched for 100 s to expose the CoFeB/MgO interface. Surprisingly, there are a lot of Ta peaks in all the samples, representing Ta⁰ 4f_{7/2}, Ta⁰ 4f_{5/2}, Ta⁵⁺ 4f_{7/2} and Ta⁵⁺ 4f_{5/2}, respectively [22]. In general, this suggests that a large amount of Ta atoms have diffused into the CoFeB layer and part of them have combined with O to form TaO_x. At the same annealing temperature of 400 °C, the amount of Ta diffusing into the interface of sample D5 (red line) is much lower than that of sample A (orange line) (Table 1), which implies that the periodically grown buffer layers have served as a diffusing barrier and suppressed the diffusion of Ta. As shown in Table 1, the annealed sample D5 has a stronger TaO_x peak intensity than the as deposited D5, suggesting that the annealing process favors the formation of TaO_x.

XPS spectra of Mg 1s are illustrated in Fig. 5c. The peak height represents the amount of Mg²⁺ in the MgO layers. Sample D5 as-deposited possesses the highest peak intensity compared to the other two annealed samples (Table 1), suggesting that MgO is partially decomposed during annealing process, resulting less Mg²⁺ left. The released elemental O are likely to combine with other metals (Ta or Fe) and form corresponding oxides. This is consistent with the observation of TaO_x in Fig. 5b. Here it is noticeable that annealed D5 sample shows higher Mg peak than annealed A sample. This can be understood as that more Ta atoms at the CoFeB/MgO interface in sample A, promotes the decomposition of MgO because of the formation of TaO_x, resulting lesser Mg²⁺ left.

Fig. 5d presents Fe 2p XPS spectra of sample A and sample D5 w/o anneal after etching for 100 s. It is obvious that a significant portion of FeO_x has been formed in sample D5 w/o anneal. To quantitatively

analyze the data, seven peaks including Fe 2p_{3/2} in metallic Fe, Fe 2p_{3/2} in FeO_x (two peaks), Fe₃O₄, the Auger peak of Co, metallic Fe 2p_{1/2}, Fe 2p_{1/2} in FeO_x have been used to fit the data [23]. And the proportion of FeO_x in the total content can be extracted as shown in Table 1. There is a small amount FeO_x formed in sample A, because there is too much Ta at the interface. It can also be seen that the annealed sample D5 have the highest FeO_x content of 43 % at the interface, accompanied by lower TaO_x peaks. This FeO_x peak is consistent with the strongest hybridization of Fe 3d and O 2p orbital, resulting the robust PMA in sample D5 [24]. It is worth noting that the addition of a small amount of FeO_x does indeed improve annealing stability, consistent with other reports [7,13].

There are Ta atoms diffused to the CoFeB/MgO interface during growth and annealing process. And because Ta (1.5) has lower electronegativity than Fe (1.83), it is easier to form TaO_x than FeO_x. As shown in Table 1, more TaO_x means lesser FeO_x, and lesser thermostability of PMA. On the other hand, with the help of the Pt/Ta superlattice buffer layer, lesser Ta can diffuse into the CoFeB/MgO interface. Thus, more FeO_x formed and enhanced the PMA.

4. Conclusion

In Summary, we have successfully demonstrated that by growing {(Pt/Ta)}_n superlattice buffer layers with $n = 3$ and $t = 1.5$ nm below the CoFeB/MgO/Ta multilayers can improve the thermostability of the films' PMA up to 440 °C, which is compatible with the standard CMOS process. The periodic structure reduces the amount of Ta from diffusing to the CoFeB/MgO interface, increases the formation of FeO_x and enhances the orbital hybridization of Fe and O, thereby improving the annealing stability of CoFeB/MgO/Ta multilayers. In conclusion, the method of periodically grown buffer layers provides a new feasible solution for enhancing CoFeB/MgO/Ta multilayers' thermostability.

See the supplementary material for MOKE and M – H images of different samples at different temperatures.

CRediT authorship contribution statement

Shuanghai Wang: Writing – review & editing, Writing – original draft, Validation, Investigation, Formal analysis, Conceptualization. **Jin Wang:** Investigation. **Xiaolong Zhang:** Investigation. **Guanqun Feng:** Investigation. **Ruobai Liu:** Resources. **Yongkang Xu:** Investigation. **Xingze Dai:** Investigation. **Kun He:** Investigation. **Yilin Zhang:** Investigation. **Yafeng Deng:** Investigation. **Yao Li:** Resources. **Yu Yan:** Resources. **Jun Du:** Resources. **Yong-Lei Wang:** Resources. **Yongbing Xu:** Resources. **Liang He:** Writing – review & editing, Resources, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.vacuum.2023.112913>.

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