Thermally robust Mo/CoFeB/MgO trilayers with strong perpendicular magnetic anisotropy

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The recent discovery of perpendicular magnetic anisotropy (PMA) at the CoFeB/MgO interface has accelerated the development of next generation high-density non-volatile memories by utilizing perpendicular magnetic tunnel junctions (p-MTJs). However, the insufficient interfacial PMA in the typical Ta/CoFeB/MgO system will not only complicate the p-MTJ optimization, but also limit the device density scalability. Moreover, the rapid decreases of PMA in Ta/CoFeB/MgO films with annealing temperature higher than 300°C will make the compatibility with CMOS integrated circuits a big problem. By replacing the Ta buffer layer with a thin Mo film, we have increased the PMA in the Ta/CoFeB/MgO structure by 20%. More importantly, the thermal stability of the perpendicularly magnetized (001)CoFeB/MgO films is greatly increased from 300°C to 425°C, making the Mo/CoFeB/MgO films attractive for a practical p-MTJ application.

Magnetic tunnel junctions (MTJs) with ferromagnetic electrodes possessing perpendicular magnetic anisotropy (PMA) have the potential for realizing high-density magnetoresistance random access memories (MRAMs)1,2. Such MTJs combine several important advantages over the MTJs with in-plane anisotropy, such as higher energy barrier against thermal agitation at reduced dimensions, smaller critical current density and faster reversal speed for current induced magnetization switching (CIMS)3–5. However, all the conventional PMA materials, including rare-earth/transition-metal alloys6–7, L12–ordered (Co, Fe)-(Pt, Pd) alloys8–9, and Co/(Pd, Pt, Ni) multilayers10–11, suffer from either insufficient chemical/thermal stability or difficulties in integrating them into MTJs. In fact, the perpendicular MTJs (p-MTJs) based on these materials exhibit a relatively low magnetoresistance ratio and a high critical current needed for CIMS due to their low spin polarization and large Gilbert damping constant6–11. The discovery of appreciable interfacial PMA in the Ta/CoFeB/MgO films together with the demonstration of high performance p-MTJs by utilizing this well developed material system is a real breakthrough7. The discovery of appreciable interfacial PMA in the Ta/CoFeB/MgO films together with the demonstration of high performance p-MTJs by utilizing this well developed material system is a real breakthrough7. Further study showed that an electric field can be used to assist magnetization switching with the electric current density reduced by two orders in the perpendicular CoFeB/MgO MTJs12. These findings represent significant steps towards the next generation spintronic devices. However, the interfacial PMA in the Ta/CoFeB/MgO system is insufficient for practical applications to some extent. It will not only limit the device density scalability, but also complicate p-MTJ optimization considering the ultrathin CoFeB layer with a very narrow tunable thickness range. Moreover, the rapid decrease of PMA in Ta/CoFeB/MgO films with the annealing temperature higher than 300°C significantly degrades the performance of the p-MTJs13–16. Since MRAMs with multi-level interconnects require a processing temperature of 350°C or higher17,18, it is urgently demanded to improve the thermal stability of the perpendicular CoFeB/MgO films. By replacing the Ta buffer with a Hf layer, we were able to raise the PMA of CoFeB/MgO films by 30% but without improvement in its thermal stability19. In this paper, we have explored the effect of Mo buffer on the magnetic properties of CoFeB/MgO films, and found that the Mo/CoFeB/MgO films can endure thermal annealing at temperatures of up to 425°C along with appreciable enhancement in the PMA.

Results
Films of Ta(5)/Co40Fe40B20(0.8–1.8)/MgO(2)/Ta(5) and Mo(5)/Co40Fe40B20(0.8–1.8)/MgO(2)/Mo(5) (in nm) annealed at a relatively low temperature of 300°C were first characterized magnetically. Figures 1(a) and (b) show the representative M–H curves of the Ta/CoFeB/MgO films with the external field perpendicular and parallel to
ing the heat treatment. Figure 2 shows the film, respectively. PMA is only observed for both Ta and Mo buffered films. The interfacial PMA in Ta/CoFeB/MgO films was measured to be about 1040 emu/cm². The large increase in perpendicular (a), (c), and parallel (b), (d) to the film plane. The numbers in the figures correspond to the CoFeB layer thickness.

The magnetic anisotropy together with appreciable moment loss. For the Ta/CoFeB/MgO films, annealing at 300°C leads to the collapse of the perpendicular magnetization in the Ta/CoFeB/MgO films larger than that of Ta buffered samples. It was proposed that the buffer layer with a high negative formation enthalpy for its boride compounds may lead to better crystallization of the CoFeB/MgO interface and thus improving Kₐ. Clearly this is not the case here, because molybdenum borides were predicted to have much smaller enthalpies in comparison with tantalum borides. The stress effect of the Mo buffer, if any, is also eliminated by the fact of Kₐ enhancement in the Ta/ultra thin Mo/CoFeB/MgO films as shown later. The PMA of Ta/CoFeB/MgO films was found to exhibit the maximum value at the annealing temperature of 250°C due to the Ta/CoFeB intermixing at higher temperatures. The little interdiffusion in Mo buffered CoFeB/MgO films, which will be proved below, likely contributes to the larger Kₐ in the Mo/CoFeB/MgO films annealed at 300°C.

We then studied the magnetic properties of the perpendicularly magnetized CoFeB/MgO films with Ta and Mo buffers at different annealing temperatures. For the Ta/CoFeB/MgO films, annealing at temperatures above 300°C leads to the collapse of the perpendicular magnetic anisotropy together with appreciable moment loss. Figure 3(a) and (b) show the M-H loops of the representative Ta/CoFeB/MgO films annealed at 325°C. Note that, the film with t_{CoFeBconst} = 1.0 nm turns to exhibit in-plane anisotropy, whereas the sample with t_{CoFeBconst} = 0.8 nm even has zero remanence with rounded in-plane and perpendicular M-H curves coinciding with each other, resembling superparamagnetism. Meanwhile, both samples show the areal saturation magnetization reduced almost by half in comparison with the 300°C annealed films [cf. inset of Fig. 2]. When the annealing temperature increases to 350°C, the Ta/CoFeB/MgO films are further deteriorated. In addition to more moment loss, all films with t_{CoFeBconst} ≤ 1.1 nm present superparamagnetic-like M-H curves (not shown). These results are in agreement with those reported
recently. Overall, the perpendicularly magnetized Ta/CoFeB/MgO films are rather unstable against annealing. It should be pointed out that, Ta interdiffusion has been demonstrated in magnetic multi-layer films after heat treatment, which not only degrades MTJs, but also causes significant moment loss and PMA deterioration, especially for the perpendicularly magnetized Ta/CoFeB/MgO films with a very thin CoFeB layer.

For the Mo/CoFeB/MgO films, the large interfacial perpendicular magnetic anisotropy remains unchanged and all trilayers with $t_{CoFeB} \leq 1.2$ nm exhibit perpendicular magnetization after annealing at temperature ($T_{an}$) up to 425°C. Figure 3(c) and (d) display M-H curves of the 425°C annealed Mo/CoFeB/MgO films with $t_{CoFeB} = 0.8$ and 1.1 nm, respectively. A square perpendicular loop is evident with remanence ratio exceeding 0.9 for both samples. The effective perpendicular magnetic anisotropy field, estimated from the in-plane M-H curve, is about 6 kOe for $t_{CoFeB} = 0.8$ nm and 1.5 kOe for $t_{CoFeB} = 1.1$ nm, comparable to the values for the 300°C annealed samples. Clearly the strong PMA for the trilayers with a thin CoFeB layer is maintained after the extremely high temperature annealing. It should be mentioned that there is no magnetic dead layer formation with the magnetization even slightly larger than that of the 300°C annealed films, which will be detailed later. Parenthetically, since the shape anisotropy increases with the increase of the magnetization, the Mo/CoFeB/MgO film with $t_{CoFeB} = 1.3$ nm after annealing at 425°C no longer shows perpendicular magnetization. In summary, by simply replacing the typical Ta buffer with a Mo layer, a thermally robust perpendicularly magnetized CoFeB/MgO system is realized. This result also suggests that there is little Mo interdiffusion during the high temperature annealing, even for the case of the extremely thin CoFeB layer. We have further studied the films of Ta/CoFeB/MgO film, the Ta/Mo(0.5)/CoFeB/MgO film with 0.8 nm $\leq t_{CoFeB} \leq 1.1$ nm presents well defined PMA at $T_{an} \leq 350$°C, indicating the significantly improved thermal stability. Figure 3(e) and (f) show M-H curves of the 350°C annealed Ta/Mo(0.5)/CoFeB/MgO films with $t_{CoFeB} = 0.8$ and 1.1 nm, respectively. The samples have large effective perpendicular magnetic anisotropy field along with the unreduced magnetization. It is likely that the serious diffusion of Ta into the thin CoFeB layer in the Ta/CoFeB/MgO films at $T_{an} = 350$°C is effectively hampered by the ultrathin Mo insertion layer, showing the crucial role of Mo as a diffusion barrier in magnetic multilayers.

Based on the M-H curves of various CoFeB thicknesses, the interface anisotropy ($K_I$), the CoFeB saturation magnetization ($M_S$), and the nominal magnetic dead layer thickness ($t_d$) were determined for both Ta/CoFeB/MgO and Mo/CoFeB/MgO films annealed at temperatures between 300–450°C. The results are summarized in Fig. 4. Note that for the Ta/CoFeB/MgO films, $K_I$ sharply decreases from 1.7 to 1.2 erg/cm² as $T_{an}$ increases from 300 to 325°C, and further falls to 0.9 erg/cm² at $T_{an} = 350$°C. Meanwhile, a magnetic dead layer that is equivalent to the observed large moment loss is given as $t_d = 0.45$ nm at $T_{an}$ = 325°C and $t_d = 0.55$ nm at $T_{an}$ = 350°C. With the dead layer part subtracted, the $M_S$ of the CoFeB layers changes minimally in this annealing temperature range. All these parameters indicate the serious deterioration of the CoFeB layer...
due to the interdiffusion at \( T_{\text{an}} > 300^\circ \text{C} \). For the Mo/CoFeB/MgO films, the large \( K_u \) is maintained within 2.05 ± 0.07 erg/cm\(^2\) at 300°C ≤ \( T_{\text{an}} \) ≤ 425°C, and there is almost no magnetic dead layer formation. The apparent \( K_u \) decrease and appreciable magnetic dead layer formation are observed at the elevated annealing temperature of 450°C. The \( M_s \) of Mo/CoFeB/MgO films slightly increases till \( T_{\text{an}} = 375^\circ \text{C} \), then it rises at a faster rate. Such increase in \( M_s \) should be caused by CoFeB crystallization improvement. In short, the Mo/CoFeB/MgO films with larger PMA are thermally stable up to 425°C. Parenthetically, the \( K_u \) of the 300°C annealed Ta/Mo(0.5)/CoFeB/MgO films was determined to be about 1.85 erg/cm\(^2\), slightly larger than that for Ta/CoFeB/MgO. This \( K_u \) value is unchanged at \( T_{\text{an}} = 350^\circ \text{C} \), but sharply decreases to 1.4 erg/cm\(^2\) at \( T_{\text{an}} = 375^\circ \text{C} \), accompanied with a dead layer increasing from 1 Å to 4 Å. This result indicates that the interfacial ultrathin Mo between Ta and CoFeB layers considerably improves PMA and the thermal stability due to the suppressed Ta interdiffusion.

Besides the thorough investigation of the CoFeB/MgO films grown on a Ta or Mo buffer layer, the films with the inverted structure, namely MgO(5)/Co\(_{40}\)Fe\(_{40}\)B\(_{20}\)(0.8–1.8)/Ta(5) or Mo(5) (in nm), were also examined. After annealing at 300°C, the Ta and Mo capped MgO/CoFeB films show perpendicular magnetization with the same upper limit thickness of 1.5 nm for the CoFeB layer in both sets of samples. However, the MgO/CoFeB/Ta films have a nominal dead layer thickness of about 0.5 nm whereas the MgO/CoFeB/Mo films only about 0.25 nm. The dead layer formation in MgO/CoFeB/Ta films was reported previously\(^3\). Clearly, this dead layer thickness is reduced in the MgO/CoFeB/Mo films. Moreover, the magnitude of \( K_u \) is about 1.6 erg/cm\(^2\) for the Ta capped films, and 1.9 erg/cm\(^2\) for the Mo capped samples. Both values are slightly lower than the results obtained in the Ta and Mo buffered samples, showing the effect of the stacking sequence. Most importantly, serious magnetic deterioration occurs in MgO/CoFeB/Ta at \( T_{\text{an}} = 325^\circ \text{C} \), but there is neither PMA worsening nor further moment loss in MgO/CoFeB/Mo when \( T_{\text{an}} \) is elevated up to 425°C. These results indicate that the MgO/CoFeB films covered by Mo also present larger PMA and significantly improved thermal stability.

Finally, in order to evaluate the suitability of the Mo-based CoFeB/MgO films for practical p-MTJs application, the multilayer films of Mo(5)/Co\(_{40}\)Fe\(_{40}\)B\(_{20}\)(0.8–1.2)/MgO(2)/Co\(_{40}\)Fe\(_{40}\)B\(_{20}\)(0.8–1.2)/Mo(5) (in nm) annealed at 400°C were characterized magnetically and structurally. First of all, strong PMA presents in all these multilayer films after high temperature annealing. Figure 5 shows M-H curves of two representative samples, Mo/CoFeB(1.2)/MgO/CoFeB(1.2)/Mo and Mo/CoFeB(0.9)/MgO/CoFeB(1.1)/Mo. Note that, perpendicular magnetization is evident even for the multilayer with the top and bottom CoFeB layers as thick as 1.2 nm. Because of the small switching field difference, the top and bottom CoFeB layers with the same thickness simultaneously switch their perpendicular magnetization via the domain nucleation mechanism. It was previously shown that, independent magnetization switches were achieved in sub-micrometer size patterned MTJs even for such a symmetric stack due to the subtle difference in individual layer microstructures and dimensions\(^5\). By adjusting the CoFeB thickness, as shown in Fig. 5(b), appreciable different switching fields for the top and bottom CoFeB layers can also be readily achieved in the asymmetric multilayer stack. In short, magnetically the multilayer structure Mo/CoFeB/MgO/CoFeB/Mo is suitable for p-MTJs with strong thermal stability.

Figure 6(a) shows the cross-section high-resolution transmission electron microscopy (HRTEM) image of the 400°C annealed Mo(5)/CoFeB(1.2)/MgO(2)/CoFeB(1.2)/Mo(5) (in nm) film. The stacking structure of different continuous layers is clear. The fast Fourier transforms (FFT) from the square area “b” across the interface is shown in Fig. 6(b), where the bcc [110] zone axis of CoFeB and the fcc [100] zone axis of MgO can be identified as the spots marked out by circles and triangles, respectively. The (002) diffraction spots of bcc CoFeB and fcc MgO are indexed in their corresponding diffraction pattern, which is along the layer stacking direction. Figure 6(c) presents the FFT diffraction spots from the “c” region of Mo layer. This pattern corresponds well with bcc [001] zone axis with (110) indexed. The planar selected area electron diffraction pattern from the same film is displayed in Fig. 6(d). The polycrystalline diffraction rings match the different layer orientation perpendicular to the film. These
CoFeB/MgO films are very promising for practical perpendicular anisotropy and negligible detrimental intermixing after two hours annealing at temperatures of up to 425°C. MgO films are highly stable. With enhanced perpendicular magnetic anisotropy that makes the perpendicularly magnetized Mo/CoFeB/MgO layered system more stable than MgO, the out-diffusion of the metastable Mo film has a crystalline structure. Since the MgO layer was deposited from a Ta(5)/Mo(0.5)/CoFeB(0.8–1.8)/MgO(2)/Ta(5) sample annealed at 400°C for two hours, the presence of MgO layer nearby. Secondly, the thin Ta film sputtered on a thermally oxidized Si substrate is amorphous, whereas the metastable amorphous structure has a higher energy, the out-diffusion of the amorphous atoms can be accelerated during annealing as observed by Yamanouchi et al.27 From the structural point of view, the interdiffusion of Ta in the Ta/CoFeB/MgO samples can be also intensified, whereas the Mo atoms stay in the stable state.

Discussion

It is known that both Tantalum and Molybdenum are highly refractory materials. Some characteristics about these two materials have to be considered to account for the significant improvement of perpendicular magnetic properties against thermal annealing in the Mo/CoFeB/MgO films. First, the Tantalum oxide with a very high negative formation enthalpy (~2046 kJ/mol) is thermodynamically more stable than MgO (~602 kJ/mol)33, which can reinforce the Ta diffusion towards the CoFeB/MgO interface during annealing. In contrast, with the formation enthalpies of Molybdenum oxides (~589 kJ/mol for MoO2 and ~745 kJ/mol for MoO3) comparable to that of MgO, the Mo atoms should be hardly affected by the presence of MgO layer nearby. Secondly, the thin Ta film sputtered on a thermally oxidized Si substrate is amorphous, whereas the sputtered Mo film has a crystalline structure. Since the metastable amorphous structure has a higher energy, the out-diffusion of the amorphous atoms can be accelerated during annealing as observed by Yamanouchi et al.27 From the structural point of view, the interdiffusion of Ta in the Ta/CoFeB/MgO samples can be also intensified, whereas the Mo atoms stay in the stable state.

In conclusion, it is the superior thermal endurance of molybdenum that makes the perpendicularly magnetized Mo/CoFeB/MgO films highly stable. With enhanced perpendicular magnetic anisotropy and negligible detrimental intermixing after two hours annealing at temperatures of up to 425°C, the Mo buffer (001)CoFeB/MgO films are very promising for practical perpendicular magnetic tunnel junction application.

Methods

Samples in this work mainly include three sets of CoFeB/MgO films with different buffer layers and one set of CoFeB/MgO/CoFeB multilayers, i.e.

1. Ta(5)/CoFeB(0.8–1.8)/MgO(2)/Ta(5) and the inverted, MgO(2)/CoFeB(0.8–1.8)/Ta(5).
2. Mo(5)/CoFeB(0.8–1.8)/MgO(2)/Mo(5) and the inverted, MgO(2)/CoFeB(0.8–1.8)/Mo(5).
3. Ta(5)/Mo(0.5)/CoFeB(0.8–1.8)/MgO(2)/Ta(5).
4. Mo(5)/CoFeB(0.8–1.8)/Mo(2)/CoFeB(0.8–1.2)/Mo(5),

where the numbers in the brackets are the nominal thickness of the individual layers in nanometer (nm). All films were deposited on the thermally oxidized Si wafers at room temperature by magnetron sputtering. The base pressure of the sputtering system was lower than 4 × 10⁻⁷ Pa and working argon pressure was 0.5 Pa. The films of Mo, Ta and CoFeB were dc sputtered from the respective Mo, Ta and CoFeB alloy targets, whereas the MgO layer was deposited from a MgO target by rf sputtering. To improve film homogeneity, up to 18 uniform films were prepared in a single deposition run. All samples were subsequently annealed at 300–450°C for two hours in vacuum (3 × 10⁻² Pa). Magnetic properties were studied using vibrating sample magnetometer (VSM). The microstructure characterization was performed on FEI Tecnai F30 scanning transmission electron microscopy (STEM). Cross-section TEM samples were prepared using a standard procedure consisting of gluing, wire saw cutting, mechanical polishing, dimpling, and ion milling. All presented results are obtained from room temperature measurements on the annealed samples unless otherwise specified.

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Author contributions
J.W.C. planned and supervised the study. T.L., Y.Z. and J.W.C. wrote the manuscript. T.L. prepared samples and carried out the magnetic property measurement. T.L., Y.Z. and H.Y.P. performed the TEM measurement. All authors analyzed the data, discussed the result and commented on the manuscript.

Additional information
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