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# Enhancement of perpendicular magnetic anisotropy and thermal stability in Co/Ni multilayers by MgO/Pt interfaces

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# MgO/Pt界面对增强Co/Ni多层膜垂直磁各向异性及热稳定性的研究*

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采用直流磁控溅射法在玻璃基片上制备了Pt底层和MgO/Pt双底层的Co/Ni多层膜样品, 通过反常霍尔效应研究了不同MgO厚度和退火温度对样品垂直磁各向异性(perpendicular magnetic anisotropy, PMA)的影响. 随着底层中MgO厚度的逐渐增加, 样品的矫顽力也随之增强, 霍尔电阻变化不大; 对样品进行退火处理后发现, 单纯Pt底层的Co/Ni多层膜随着退火温度的升高, 霍尔电阻逐渐降低, 矫顽力则迅速降低, 热稳定性较差; 而当MgO/Pt双底层的样品在200 °C退火后矫顽力大幅增加, 霍尔电阻略微有所减小, 更高的退火温度使得Co和Ni合金化, 导致多层膜的PMA特征减弱.

**关键词:** 磁性多层膜, 垂直磁各向异性, 反常霍尔效应

**PACS:** 75.70.–i, 75.30.Gw, 75.47.–m **DOI:** 10.7498/aps.64.197501

## 1 引 言

自旋转移力矩效应在研究磁随机存储器(spin-transfer-torque magnetoresistive random access memory, STT-MRAM), 自旋极化电流激发的静态和动态磁性状态等方面有着重要应用[1], 基于自旋转移力矩的磁随机存储器由于读写时间短、存储容量大、尺寸更小等优势而非常具有发展潜力, 具有垂直磁各向异性(perpendicular magnetic anisotropy, PMA)的磁纳米多层膜是STT-MRAM至关重要的组成部分[2], 该类材料需要具备良好的热稳定性和较低饱和场, 以便降低垂直磁纳米结构电流诱导磁化翻转的电流密度, 从而降低器件功耗[3]. 对此类材料的研究大都集中铁磁层/非磁层结构中, 如Co/Pt, CoFeB/Pt[4,5]等多层膜结构.

由于Co和诸如Pt, Pd, Au及Ni等金属层之间有较强的界面各向异性能, 所以经常被用来制备具有PMA性质的多层膜[6−9], 相比其他垂直多层膜结构, Co/Ni多层膜由于具备更大的巨磁电阻效应和较低的单轴各向异性而受到关注[10−12]. 一般地, 可以通过调整多层膜的底层结构来增强多层膜的界面散射, 从而提高多层膜的PMA, 但目前对多层膜底层的研究大都集中在金属界面之间. 非晶态的绝缘层/金属层界面电子的附加散射, 可以使多层膜的PMA性能提高[13], 这在隧道磁电阻和各向异性磁电阻材料方面有着重要应用[14,15]. MgO作为绝缘层, 被用在Co/Pt多层膜的底层中, 使得多层膜获得了比单纯Pt作为底层更为优异的PMA性质[16], 而在Co/Ni多层膜中应用MgO作为底层的报道很少. 此外, 对单纯金属底层Co/Ni多层膜PMA的热稳定性有一定的研究[17], 对于MgO为底层Co/Ni多层膜PMA的热稳定性研究还少见报道.

本文应用磁控溅射法, 制备了以Pt和MgO/Pt为底层的Co/Ni多层膜, 并对其进行了退火处理,

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通过反常霍尔效应方法研究不同MgO厚度、不同退火温度对样品PMA性质的影响, 以期通过在Co/Ni多层膜底层中加入适当厚度MgO和适当温度退火处理增强样品的PMA.

## 2 实 验

所有样品均采用磁控溅射法在玻璃基片上制备而成, 设备工作时样品台以1.7 r/s的速度自转, 以便获得生长均匀的样品. 溅射系统本底真空度优于$2.0\times10^{-5}$ Pa, 工作气体为Ar气(99.999%), 工作气压为0.5 Pa. 靶材的溅射速率由Dektak150型台阶仪测定, 分别为MgO: 0.035 nm/s, Pt: 0.075 nm/s, Co: 0.047 nm/s, Ni: 0.042 nm/s. 样品结构分别为Pt(2)/ Co(0.2)/ Ni(0.4)/Co(0.2)/ Pt(2)和MgO(t)/ Pt(2)/ Co(0.2)/ Ni(0.4)/Co(0.2)/ Pt(2), 本文中样品厚度均用nm表示, 其中MgO厚度$t_{\mathrm{MgO}}$的变化范围从1到5, 所有样品用2 nm厚Pt做保护层防止样品氧化. 对制备好的样品在实验室自制的退火炉中进行退火, 腔体的真空度优于$2.0\times10^{-5}$ Pa.

磁性材料的霍尔电阻率$\rho_{xy}$与外加磁场$B$的关系为[18]

$$\rho_{xy}=R_{\mathrm{o}}B+4\pi R_{\mathrm{S}}M_{\perp}, \tag{1}$$

其中$R_{\mathrm{o}}$为正常霍尔系数, $R_{\mathrm{S}}$为反常霍尔系数, 且反常霍尔效应比正常霍尔效应大一个数量级以上, 一般认为$\rho_{xy}$正比于样品磁矩的垂直分量[19], 可以通过测量样品的霍尔回线来研究磁性薄膜的PMA性质. 样品的霍尔回线用四探针法进行测量, 可以获取样品的霍尔电阻(Hall resistance, $R_{\mathrm{Hall}}$)和矫顽力(coercivity, $H_{\mathrm{C}}$)等信息, 磁场方向垂直于膜面. 由国家纳米科学中心综合物性测试系统(PPMS)中的振动样品磁强计(VSM)组件测量样品的磁滞回线.

## 3 实验结果与讨论

我们对Co/Ni多层膜PMA的前期研究表明: 通过调制各种参数可以获得以Pt为底层的具有良好PMA的样品Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/Pt(2)[20], 为了研究样品的热稳定性, 对其退火后的霍尔回线进行了测量, 如图1(a)所示, 图1(b)为样品霍尔电阻及矫顽力随退火温度的变化. 可以看到样品在100 °C退火后剩磁比及矩形度保持的很好, 霍尔电阻较未退火时有所降低但不多, 退火温度高于100 °C时, 样品矫顽力迅速减小, 霍尔电阻也随之大幅度的降低, 可见该样品的热稳定性不是很好, 退火并未增强样品的PMA.

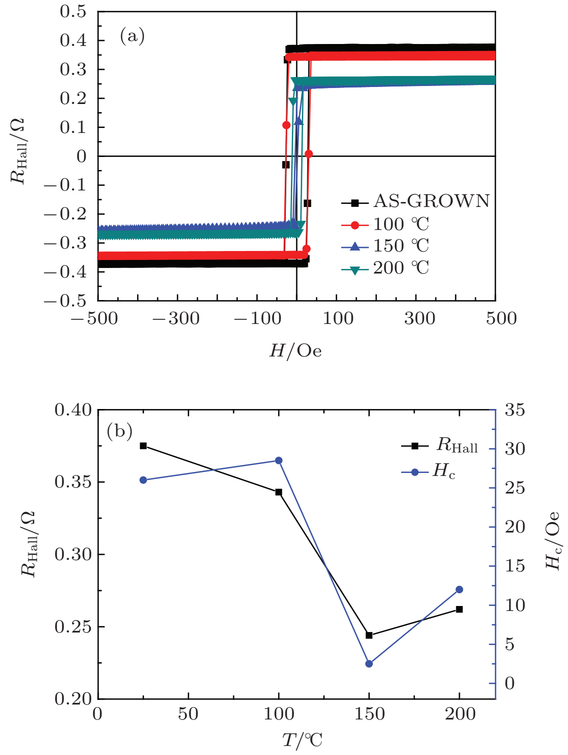

图1 (网刊彩色) (a) 退火后Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/ Pt(2) 的霍尔回线 (1 Oe = 79.5775 A/m); (b) 退火后Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/Pt(2) 的霍尔电阻及矫顽力

Fig. 1. (color online) (a) Annealing temperature dependence of Hall loops for Pt(2)/Co(0.2)/Ni(0.4)/ Co(0.2)/Pt(2); (b) annealing temperature dependence of $R_{\mathrm{Hall}}$ and $H_{\mathrm{C}}$ for Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/Pt(2).

为了获得更好PMA性质的Co/Ni多层膜样品, 制备了一系列以MgO/Pt为底层的样品, 这样可以增加非晶态的绝缘层/金属层界面, 以期利用该界面较强的电子附加散射来增强样品的PMA性质. 图2(a)为样品MgO(t)/Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/Pt(2)的霍尔曲线, 可以看到, 在MgO底层逐渐变厚的过程中, 所有样品的磁矩翻转过程均很迅速, 矩形度保持的非常好, 且样品的剩磁比均达到了100%, 说明样品具有良好的PMA性质. 图2(b)为其霍尔电阻及矫顽力随MgO底层厚度$t_{\mathrm{MgO}}$的变化曲线, 可以看到样品的霍尔电阻随着MgO的厚度在一定的范围内小幅度波动, 可见

MgO的加入对样品的霍尔电阻影响不大, 这是因为作为氧化层, MgO的分流作用很弱, 所以样品的霍尔电阻并未有明显变化. 而矫顽力却随着MgO底层的逐渐变厚出现了大幅度的增加, 当MgO为1 nm时, 样品矫顽力较单纯Pt底层有所增加, 可见此时形成的绝缘层/金属层界面对样品的性能有了一定的影响, 但由于MgO厚度较薄, 首先要克服基片本身的不平整对界面造成的影响, 随着MgO厚度的逐渐增加, MgO/Pt界面变得更为平整, 一方面, 在MgO上面生长的Pt更易形成良好的(111)织构, 从而引导Co/Ni多层膜的(111)织构, 所以样品的矫顽力迅速增加; 另一方面MgO/Pt界面随着MgO厚度的增加, 使得界面的电子附加散射更为明显, 样品性能比单纯Pt底层时更为优异, 从而获得了良好的PMA性质. 当MgO厚度为4 nm时, 样品的矫顽力达到了最大值, 较单纯Pt底层时增加了约2.3倍, 且样品保持了非常好的矩形度, 样品的霍尔电阻较不加MgO底层时增加约9%.

为了反映底层加入MgO对样品表面及界面平整度的影响, 分别将样品Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/Pt(2)及样品MgO(t)/Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/Pt(2)进行了AFM测试, 如图3所示. 经测算, 粗糙度分别为0.192 nm和0.115 nm, 可见加入4 nm厚MgO底层后, 大幅度的降低了样品的粗糙度, 结构方面的变化对样品的性能产生了影响, 这和图2中的测试结果相吻合. 为了研究MgO/Pt界面的加入对样品热稳定性的影响, 对样品MgO(4)/ Pt(2)/ Co(0.2)/ Ni(0.4)/ Co(0.2)/ Pt(2)进行退火处理.

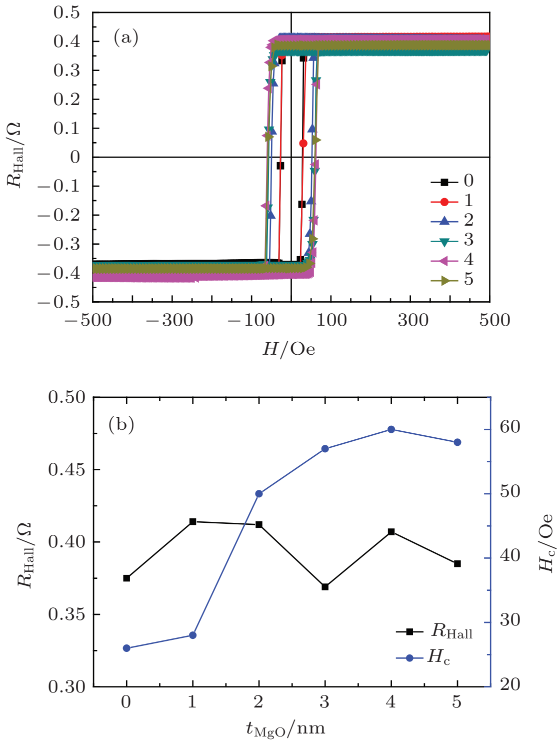

图2 (网刊彩色) (a) MgO(t)/Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/ Pt(2)的霍尔曲线; (b) MgO(t)/Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/ Pt(2)的霍尔电阻及矫顽力

Fig. 2. (color online) (a) Hall loops of MgO(t)/Pt(2)/Co(0.2)/ Ni(0.4)/ Co(0.2)/Pt(2); (b) $R_{\mathrm{Hall}}$ and $H_{\mathrm{C}}$ of MgO(t)/ Pt(2)/Co(0.2)/Ni(0.4)/ Co(0.2)/Pt(2).

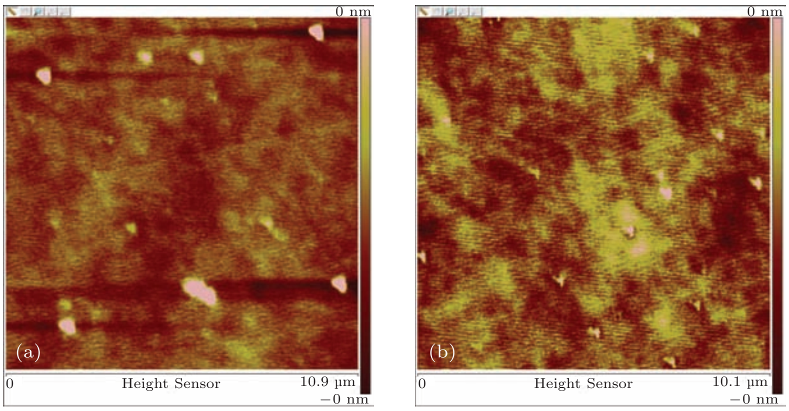

图3 (网刊彩色) Co/Ni多层膜的AFM图 (a) Pt(2)底层; (b) MgO(4)/Pt(2)底层

Fig. 3. (color online) AFM of Co/Ni multilayers: (a) Pt(2) underlayer; (b) MgO(4)/Pt(2) underlayer.

图4(a)为对样品MgO(4)/Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/Pt(2)在不同温度下退火后测得的霍尔回线, 图4(b)是样品的霍尔电阻及矫顽力随不同退火温度的变化曲线. 可以看到, 当退火温度$\leqslant$200 °C时, 样品霍尔回线的具有良好的矩形度, 剩磁比也保持在100%, 说明在这个温度范

围内, 样品保持了良好的PMA特性. 样品的矫顽力也随着退火温度的升高有着明显的增大, 当退火温度为200 °C, 样品的矫顽力达到最大值, 是未退火时的1.5倍多, 比没有MgO底层的样品更是增大了3.5倍多, 这和退火使得多层膜的界面变得更为明晰有关, 在合适的温度对样品进行退火使得界面处的元素进行重组进而从生长时的无序变为有序[18], 联系图1 (a)的实验结果, 可以认为对样品PMA性能的提升主要来自于MgO/Pt界面. 样品经200 °C退火后, 霍尔电阻较未退火状态有约6%的减小. 随着退火温度的继续升高, 样品的PMA迅速降低, 当退火温度为400 °C时, 样品失去了PMA性质, 这和过高的退火温度造成多层膜Co/Ni界面的合金化有关, 样品失去了层间耦合效应, 导致PMA的消失.

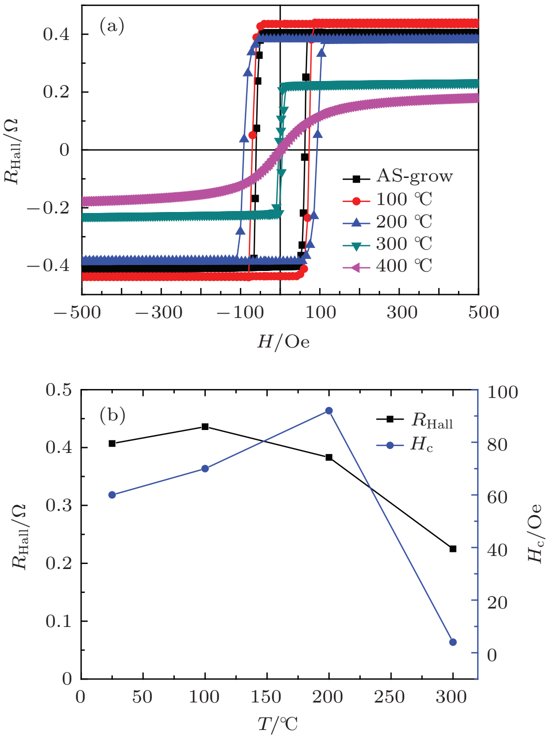

图4 (网刊彩色) (a) 退火后MgO(4)/Pt(2)/Co(0.2)/Ni(0.4)/ Co(0.2)/ Pt(2) 的霍尔曲线; (b) 退火后MgO(4)/ Pt(2)/Co(0.2)/ Ni(0.4)/ Co(0.2)/Pt(2)的霍尔电阻及矫顽力

Fig. 4. (color online) (a) Annealing temperature dependence of Hall loops for MgO(4)/Pt(2)/ Co(0.2)/Ni(0.4)/ Co(0.2)/Pt(2); (b) annealing temperature dependence of $\rm R_{Hall}$ and $H_{\rm C}$ for MgO(4)/Pt(2)/ Co(0.2)/ Ni(0.4)/Co(0.2)/Pt(2).

磁性多层膜的有效磁各向异性$K_{\rm eff}$表达式如下:

$$K_{\rm eff} = K_{\rm v} + 2K_{\rm s}/t, \tag{2}$$

式中$K_{\rm v}$和$K_{\rm s}$为磁性层的体及界面各向异性, $t$为磁性层的厚度[21]. 一般来讲由于退磁场原因$K_{\rm v} < 0$, 而界面各向异性$K_{\rm s} > 0$, $K_{\rm eff}$是体各向异性和界面各向异性竞争的结果, 若$K_{\rm eff} > 0$, 则薄膜具有PMA特征. 图5为磁场平行膜面测得200 °C退火后 MgO(4)/Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/Pt(2)归一化后的磁滞回线. 可以看出, 磁滞回线通过原点, 饱和磁场达到了$1.0 \times 10^4$ Oe, 具备典型的难轴特征. 经过计算, 样品的$K_{\rm eff}$为$8.2 \times 10^6$ erg/cm$^3$, 比单纯Pt底层Co/Ni多层膜增加了15%. 在Co/Ni 多层膜中加入MgO底层, 并未改变样品磁性层的厚度, 而样品的PMA却有极大的改善, 可见由于MgO/Pt界面的存在, 增强了Co/Ni多层膜的界面各向异性能, 而合适温度的退火, 使MgO/Pt界面的的作用更加明显, 样品表现出了良好的PMA性质.

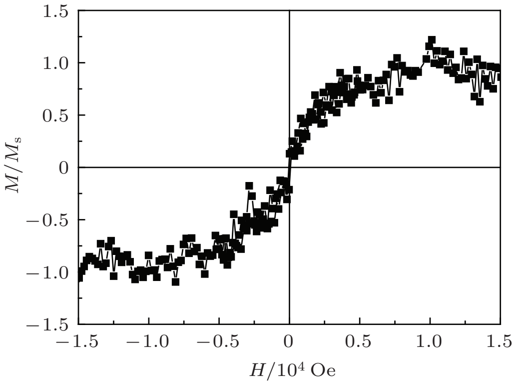

图5 200 °C退火后MgO(4)/Pt(2)/Co(0.2)/Ni(0.4)/ Co(0.2)/Pt(2)磁场平行膜面归一化的磁滞回线

Fig. 5. Normalized hysteresis loops of MgO(4)/ Pt(2)/Co(0.2)/Ni(0.4)/Co(0.2)/Pt(2) annealing at 200 °C with field applied in plane.

## 4 结 论

通过对以Pt为底层的Co/Ni多层膜中加入氧化层MgO, 一方面, 合适的MgO厚度可以减弱基片对Pt层平整度的影响, 使得Pt能够获得更好的(111)织构, 从而引导Co/Ni多层膜的(111)织构; 另一方面在多层膜中增加了绝缘层/金属层界面, 利用MgO/Pt界面的电子附加散射来增强样品的PMA性质. 通过对样品退火后磁性能的研究发现, 单纯Pt底层样品热稳定性较差, 而加入适当厚度MgO的样品可保持一定的热稳定性; 最终通过在Co/Ni多层膜中加入MgO/Pt界面并进行退火处理, 样品的PMA得到了极大的提高, 此外还需进一

步研究对 MgO/Pt 底层的预处理, 以使 Co/Ni 多层膜获得更好的 (111) 织构.

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# Enhancement of perpendicular magnetic anisotropy and thermal stability in Co/Ni multilayers by MgO/Pt interfaces[*]

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## Abstract

Co/Ni multilayers with Pt and MgO/Pt underlayer have been grown by means of magnetron sputtering and the perpendicular magnetic anisotropy (PMA) of the samples is studied using anomalous Hall effect (AHE). The Co/Ni multilayer has to be thermally stable to stabilize the PMA, which is studied by annealing treatment. In early researches of Co/Ni multilayes, the optimum sample with Pt underlayer was obtained as Pt(2 nm)/Co(0.2 nm)/Ni(0.4 nm)/Co(0.2 nm)/Pt(2 nm) with PMA in good performance. Thermal stability of the sample is studied in this paper by the Hall loop measurement of it after annealing. Results show that the remanence ratio and rectangular degree of the sample are kept well and the Hall resistance ($R_{\mathrm{Hall}}$) has little change at the annealing temperature of 100 °C. As the annealing temperature rising above 100 °C, the PMA of Pt(2 nm)/Co(0.2 nm)/Ni(0.4 nm)/Co(0.2 nm)/Pt(2 nm) becomes weakened. Its coercivity ($H_{\mathrm{c}}$) decreases rapidly and $R_{\mathrm{Hall}}$ reduces greatly. So the thermal stability of Pt(2 nm)/Co(0.2 nm)/Ni(0.4 nm)/Co(0.2 nm)/Pt(2 nm) will be poor and the PMA cannot be enhanced by annealing treatment. A series of samples with MgO/Pt underlayer are prepared with the thickness of Pt being fixed at 2 nm and that of MgO ranging from 1 to 5 nm. Thus the interface between amorphous insulation layer and metal layer is added to be used to enhance the PMA of the sample for the strong electron additive scattering. Magnetization reversal can be very rapid and the rectangular degree is kept very well, and furthermore, the remanence ratio of the samples can reach 100% so they all show good PMA.

The $H_{\mathrm{c}}$ increases with increasing MgO underlayer and reaches the maximum value as the MgO thickness arrives at 4 nm, and the $H_{\mathrm{c}}$ of the sample MgO(4 nm)/Pt(2 nm)/Co(0.2 nm)/Ni(0.4 nm)/Co(0.2 nm)/Pt(2 nm) is 2.3 times that of Pt(2 nm)/Co(0.2 nm)/Ni(0.4 nm)/Co(0.2 nm)/Pt(2 nm), the $R_{\mathrm{Hall}}$ is up to 9% correspondingly. The roughnesses of Pt(2 nm)/Co(0.2 nm)/ Ni(0.4 nm)/Co(0.2 nm)/Pt(2 nm) and MgO(4 nm)/Pt(2 nm)/Co(0.2 nm) /Ni(0.4 nm)/Co(0.2 nm)/Pt(2 nm) are 0.192 nm and 0.115 nm respectively, as tested by AFM. Result shows that the roughness of the Co/Ni multilayer is greatly reduced so the PMA of the Co/Ni multilayer is enhanced remarkably after the addition of 4 nm MgO. The thermal stability of MgO(4 nm)/Pt(2 nm)/Co(0.2 nm)/Ni(0.4 nm)/Co(0.2 nm)/Pt(2 nm) is also studied. When the annealing temperature rises up to 200 °C, the $H_{\mathrm{c}}$ reaches its maximum value i.e. 1.5 times that of the sample without MgO, and it is 3.5 times that of the sample with Pt underlayer only. This sample also show good thermal stability. Higher temperatures will result in intermixing of Co and Ni and diminish the PMA. After annealing at 400 °C, the easy axis of the sample becomes in-plane. The anisotropy constant $K_{\mathrm{eff}}$ of MgO(4 nm)/Pt(2 nm)/Co(0.2 nm)/Ni(0.4 nm)/Co(0.2 nm)/Pt(2 nm) is $8.2 \times 10^6$ erg/cm$^3$, and it has an increase of 15% in Pt(2 nm)/Co(0.2 nm)/ Ni(0.4 nm)/Co(0.2 nm)/Pt(2 nm), which shows that the sample has an excellent PMA.

**Keywords:** magnetic multilayers, perpendicular magnetic anisotropy, anomalous Hall effect

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