Temperature dependence of exchange bias in Co/FeMn-structure induced by heating and cooling in magnetic field

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Abstract. Using the method of angular dependence of ferromagnetic resonance field the magnetic properties of Si/SiO₂/Cu/Co/FeMn/Cu and Si/SiO₂/Cu/Co/Cu/FeMn/Cu structures were investigated. The layer deposition was carried out by magnetron sputtering in absence of an external magnetic field. It was established that thermal annealing with further cooling down in presence of a magnetic field can generate an exchange bias at anneal temperature significantly below the bulk antiferromagnetic Néel temperature. It was also shown that a thin interlayer between ferromagnetic and antiferromagnetic layers reduces the exchange bias effect at low anneal temperatures, however, makes this effect more stable at high annealing temperatures.

1. Introduction
A common method to generate the exchange bias in ferromagnetic (F)/antiferromagnetic (AF) thin film structures is to anneal the sample above the Néel temperature of the antiferromagnet and below the ferromagnetic Curie point with subsequent cooling down in presence of a magnetic field high enough to saturate the F-layer and applied in the plane of the sample. With such a treatment the magnetic anisotropy direction can be induced both in F and AF layers.

Recently, a number of exchange bias investigations were carried out using Irₓ₀Mn₈₀ as an antiferromagnetic layer (see for instance [1]. This alloy in contact with F layer demonstrates high exchange bias and blocking temperature values. Another prospective material for spinvalve devices is FeMn alloy with approximately 50% elements composition (see review [2]). FeMn is much cheaper material than IrMn, but characterized by lower exchange bias, blocking temperature. Néel temperature, and melting temperature values [3] and more susceptible to thermal effects, serving a convenient sample to investigate the fundamental aspects of temperature dependence of F/AF exchange biasing in a fairly narrow range near the room temperature.

In this paper the results of investigation of exchange bias generation by thermal annealing-cooling in magnetic field are reported. It was founded that in case of 7 nm AF layer thickness the exchange bias does not arise. At the thickness of 15 nm the exchange bias appears at the annealing temperature as low as T_{an}=50°C. This temperature value is much lower than the Néel temperature for the bulk
Fe$_{50}$Mn$_{50}$ ($T_N = 490$ K [2]). The annealing temperature effects on the exchange bias field and intrinsic FMR field is reported as well as influence of a thin interlayer between F and AF layers on these parameters.

2. Experimental techniques

Experimental samples were deposited by magnetron sputtering using the magnetron ATC ORION-5 produced by AJA INTERNATIONAL. The chamber was vacuumed to the base pressure of $10^{-7}$ Torr and the DC and RF depositions of the layer structures Si/Cu(50nm)/Co(5nm)/FeMn($t_{AF}$/Cu(50nm)) were made in the argon atmosphere at the pressure of $3\times10^{-3}$ Torr on Si (100) substrate with the AF-layer thicknesses $t_{AF}$ of 7 nm and 15 nm. There were also deposited the Si/Cu50nm/Co5nm/Cu0,5nm/FeMn20nm/Cu100nm structures with thin Cu interlayer. The deposition rates for each layer were evaluated by measuring the thickness of the calibrating layers using the Rutherford backscattering. Prior the deposition the Si substrates were cleaned in ethanol in an ultrasonic bath. The deposited samples were thermally annealed at $T_{ann}$ = 50, 100, 150, 200 and 250°C for 0.5 hour and cooled down to the room temperature all in presence of a magnetic field of the order about 1 kOe applied in the plane of the sample.

Magnetic properties of the samples were studied by measuring the angular dependence of the ferromagnetic resonance (FMR) field. The FMR DC magnetic field for a ferromagnetic sample, $H_{R}$, is determined by the FMR frequency $\omega = 2\pi f$ of the RF field of the spectrometer, as well as by internal magnetic properties of the sample, such as the saturation magnetization, $I_s$, magnetic anisotropy field, $H_K$, and sample orientation relative to the DC and RF fields directions. For the sample aligned with the easy axis (EA) and hard axis (HA) along the DC field the Kittel relations applied [4]:

$$\omega^2 = \gamma^2 (H_{R}^{EA} + H_k)(H_{R}^{EA} + H_k + 4\pi I_s)$$  

$$\omega^2 = \gamma^2 (H_{R}^{HA} - H_k)(H_{R}^{HA} - H_k + 4\pi I_s)$$

where, $\gamma=g\epsilon/(2mc) \approx 8.79\times10^6$ (G s)$^{-1}$ is the gyromagnetic constant, $H_{R}^{EA}$ and $H_{R}^{HA}$ are the resonance fields for EA and HA sample alignment, respectively. The Kittel relations can be generalized to include the unidirectional exchange bias field [5, 6, 7, 8]. Assuming that $\theta$ is the angle between the FMR DC magnetic field and the unidirectional exchange bias field, which is parallel to the EA of the F-layer, and for $4\pi I_s >> H_{EB}$, the resonance field can be written approximately as [Bład! Nie zdefiniowano zakładki.5; Bład! Nie zdefiniowano zakładki.6]:

$$H_{r} = \frac{\omega^2}{4\pi I_s} - H_{EB} \cos \theta - H_{K} \cos 2\theta$$

Though FMR is rather unordinary method to investigate the exchange bias phenomenon it has a high sensitivity to low magnetic moments of the FM, which can be obtained only on the most advanced VSM devices, and has an advantage in investigation the magnetic properties of structures with F layer covered by insulating, diamagnetic or antiferromagnetic layers, which have their restrictions in using other methods like MOKE or SQUID. In addition, the FMR can give the value of the sample saturation magnetization regardless to its porosity, in contrast to the VSM. In this report, from the angular dependence of the resonance field the unidirectional and uniaxial anisotropies were determined using equation (2). The measurements were done at room temperature, using BRUKER ELEXSYS e500 spectrometer with the frequency of the RF field of 9.65 GHz and the rectangular resonator of E$_{102}$ mode.

3. Experimental results and discussion

The typical differential FMR absorption spectra are shown in figure 1 for a structure with non-zero exchange bias at several sample orientation relative to the FMR DC field which was set in the sample plane.
From the resonance field, $H_r$, using equation (2) one can determine $H_{r0}, H_K, H_{EB}$ values. For this it is enough to measure the FMR spectra for three orientations, for example for $\theta = 0, 90$ or 270 and 180°.

The investigation of the structures with 7 nm AF thickness did not reveal any exchange bias at the annealing temperature in the range used. The exchange bias dependence on the annealing temperature for structures with $t_{AF} = 15$ nm is shown in figure 2. The exchange bias field showed up at $T_{ann} = 50^\circ C$. In other words, starting from this annealing temperature FeMn layer begins to arrange a certain predominating antiferromagnetic order at the AF/F interface and pins the F-layer. We note that this temperature is much lower that the Néel temperature [2], the issue to be discussed later on. At higher annealing temperatures $H_{EB}$ increases to reach the maximum value of 180 Oe at $T_{ann} = 150^\circ C$ then falls down and zeroes at $T_{ann} = 250^\circ C$.

The reduction of the $H_{EB}$ at the $T_{ann}$ above 150^\circ C is a consequence of weakening of the F and AF layers interaction. It is, probably, due to intensification of interdiffusion and layer intermixing processes on the interface at higher temperatures. An occurrence of these effects is also indicated by an FMR signal weakening and increasing of signal-to-noise ratio at these annealing temperatures. Possibly, the Mn atoms became more mobile and penetrate into Co layer causing a weakening of the ferromagnetic properties of Co layer by reducing the volume fraction of ferromagnetic Co. The FMR signal weakening was observed in the structures with 7 nm AF thickness where the interdiffusion effect can be more essential because of smaller thickness. It can also explain the absence of the exchange bias in the structures with AF layer of 7 nm reported above.

From the other hand the absence of exchange bias in these structures might result from AF small thickness. Following the reference [9], the reduction of the Néel temperature can be expressed as

$$\frac{T_N(\infty) - T_N(t_{AF})}{T_N(\infty)} = (\frac{\xi_0}{t_{AF}})^\lambda$$

where $T_N(\infty)$ and $T_N(t_{AF})$ are the Néel temperatures for the bulk material and for a film with the thickness $t_{AF}$, respectively. Assuming $\xi_0 = 3.04$ nm, $\lambda = 1.5$ as in [3] one could obtain $\Delta T_N = (T_N(\infty) - T_N(t_{AF})) \approx 140^\circ$, or $T_N(t_{AF}) \approx 350$ K for the film of 7 nm. It is a significant reduction of the Néel temperature, but could be even more, because of uncertainties in the parameters used. If the Néel point lowers down to or below the room temperature than the AF layer stays in paramagnetic state and did not pin the F-layer, which is, possibly, the case with 7 nm AF thickness.
The influence of the thermal effects on the intrinsic resonance field, $H_{r0}$, is illustrated on figure 3. Puzzling enough, the annealing temperature dependence of $H_{r0}$ has an opposite tendency compared with that obtained earlier with IrMn AF layer. In [10] the intrinsic resonance field increased at increasing $T_{\text{ann}}$ and always remained above the $H_{r0}$ for the free (i.e. not pinned) Co layer which is about 600 Oe (see accompanying paper [11]). In case of FeMn the intrinsic resonance field decreases with the temperature and tends to the value for the free Co layer. Keeping in mind the inverse dependence of $H_{r0}$ on $I_s$, one can conclude that up to 150°C the magnetization of Co layer does not change significantly being lower than that in Co free layer. At a higher $T_{\text{ann}}$ the $H_{r0}$ starts to decrease more steeply indicating an increase of $I_s$ still keeping it at a lower value compared to that in Co unpinned layer. Evidently, the origin of such a behavior of the intrinsic resonance field is complicated and not completely clear, however, the trend to the intrinsic value for unpinned Co layer is an indication of a weakening of the antiferromagnetic properties of FeMn layer with an increase of $T_{\text{ann}}$, while the magnetization of Co layer does not drastically change or even improve.

![Figure 2](image)

**Figure 2.** The dependence of exchange bias on the anneal temperature for Co/FeMn and Co/Cu/FeMn structures

In order to prevent the thermal interdiffusion and intermixing, a set of trilayer structures with thin Cu interlayer between F and AF layers was made. As can be seen from figure 2 the presence of the interlayer leads to the increase of the annealing temperature necessary to generate the exchange bias. At $T_{\text{ann}} = 50°C$ the exchange bias field is missed and appears only at $T_{\text{ann}} = 100°C$. An exponential decay of the interaction between F and AF layers and, consequently, the exchange bias with the spacer thickness was observed before [12]. The positive influence of the spacer is that it provides a certain thermal stability of the magnetic properties of the structure. As illustrated in figure 2, the exchange bias for the structure with interlayer is even higher than that one without interlayer at 200°C annealing temperature.

The intrinsic resonance field decreases in the F/Cu/AF structures with the annealing temperature increase as it does in case of the F/AF structures, see figure 3. At $T_{\text{ann}} = 100°C$ the $H_{r0}$ for the trilayer structure is lower than that for bilayer structure that means that the magnetization of F layer in the trilayer is higher. At the subsequent increasing of the anneal temperature the intrinsic resonance field value decreases more smoothly in the trilayer system that demonstrates a higher thermal stability than that of the bilayer structure.
4. Conclusions

The results of this paper show that the exchange bias at the room measuring temperature in the bilayer system Co/FeMn either can not be generated by heating-cooling in magnetic field at any temperature for thinner AF layer thickness (7 nm) or can be induced at the annealing temperature significantly lower than the bulk AF Néel temperature for larger AF layer thickness (15 nm). We conclude that this effect originates from a combination of two major sources. The first one is the low-dimensional reduction of the Néel temperature due to small film thickness, in accordance with [9]. The second reason is the interdiffusion of the layers in contacts which changes the composition with reduction of the Néel and blocking temperatures of the AF layer and lowering the saturation magnetization of the F layers, making the intrinsic FMR DC field higher. Both, the interdiffusion and the low-dimensional effects bring stronger consequences for the thinner AF films.

In accordance with other publications, see for instance [12], we observe that the nonmagnetic interlayer deposited between F and AF layers reduces the exchange bias at low anneal temperatures, however, makes this effect more stable at high anneal temperatures. This spacer weakens the interaction strength between F and AF layers. At low annealing temperature the F/AF interaction is too weak and only a small fraction of the FeMn grains with a relatively small size is set in the antiferromagnetic order. This fraction has too small unidirectional anisotropy energy to withstand the thermal fluctuations to keep the antiferromagnetic order in the AF layer and to keep the F-layer pinned at room temperature of the FMR measurements, showing no exchange bias at $T_{\text{ann}} = 50\,^\circ\text{C}$ in figure 2. However, a weak interdiffusion in the trilayer structure keeps unchanged $H_{r0}$ and $I_s$ of the Co-layer up to 200°C and making $I_s$ larger compared with the bilayer structure.

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