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Enhanced exchange anisotropy of Ni–Fe/Mn–Ni bilayers fabricated under the extremely clean sputtering process

Masakiyo Tsunoda,[a] Kazuhiro Uneyama, Toshihiro Suzuki, Kojiro Yagami, and Migaku Takahashi
Department of Electronic Engineering, Tohoku University, Sendai 980-8579, Japan

In order to clarify the influence of the impurities in the sputtering atmosphere on the exchange anisotropy of ferromagnet/antiferromagnet bilayers, Ni–Fe/Mn–Ni films were prepared under different purities of the sputtering atmosphere by changing the base pressure from 10^{-11} Torr [extremely clean (XC) process] to 10^{-7} Torr [lower grade (LG) process]. The correlation between the exchange anisotropy and the microstructure of the films is discussed. As a result, we found that: (1) The exchange anisotropy was enhanced in the XC processed films comparing to the LG processed ones, especially when the thicknesses of both the ferromagnetic and antiferromagnetic layers were very thin. (2) The critical thicknesses of the antiferromagnetic layers were 110 and 150 Å for the XC and the LG processed films, respectively. (3) In the XC processed films, the fcc-[111] direction of the Ni–Fe grains were highly oriented perpendicularly to the film plane and an enlargement of antiferromagnetic grains was observed. We conclude that the enhancement of exchange anisotropy is caused by the enlargement of antiferromagnetic grains in the XC processed films. © *1999 American Institute of Physics.* [S0021-8979(99)74708-0]

I. INTRODUCTION

The exchange biasing of a ferromagnetic (F) layer by an antiferromagnetic (AF) layer[1,2] is one of the key factors in development of the spin-valve[3] giant magnetoresistance (GMR) head for hard disk drives. Since the film constitution of spin valves should be very thin to attain a narrow shielded gap for high linear recording density, it becomes more and more important to control the microstructure of films to induce the exchange anisotropy sufficiently even in very thin F/AF bilayers. Although the microscopic origin of the exchange biasing effect has remained a subject of debate,[4,5] the strength of the exchange anisotropy is known to change easily by changing the fabrication conditions and film constitutions.[6,7] The effect of impurities is also a parameter which needs to be controlled, because impurities in the sputtering atmosphere are easily trapped in films and change the microstructure of the films by preventing epitaxial growth,[8] segregation,[9] precipitation,[10] etc. Therefore, it is important to understand and to control the role of impurities on the growth stage of thin-film devices. However, only a few attempts are reported for the effect of impurities on the spin valves, which are concerned with the vanishing of the exchange anisotropy caused by the adsorption of impurities at the F/AF interface[8] and the enhancement of the GMR ratio related to the flattening of the stacking structures.[11] In the present study, in order to make clear the influence of the impurities introduced during the deposition process on the exchange anisotropy of F/AF bilayers, we prepared Ni–Fe/Mn–Ni films by changing the purity of the sputtering atmosphere and investigated the correlation between the microstructure and the exchange anisotropy.

[a]Electronic mail: tsunoda@ecei.tohoku.ac.jp

II. EXPERIMENTAL PROCEDURE

Substrate/Ta 50 Å/Ni–Fe d_F/$Mn_{0.71}Ni_{0.29}$ d_{AF}/Ni–Fe 1.5 $\times d_F$ quadrilayered films were prepared at room temperature on a Si wafer with/without a thermally oxidized layer by a specialized rf magnetron sputtering machine made of aluminum alloys which enables a vacuum as low as 8×10^{-12} Torr. The thickness of the Ni–Fe layer, d_F, and the thickness of the Mn–Ni layer, d_{AF}, were varied. The top Ni–Fe layer was prepared just as a protection for the Mn–Ni layer from oxidation. In this article we are concerned only with the Ni–Fe layer on the substrate side in as-deposited films. A magnetic field of 30 Oe parallel to the substrate surface was applied during deposition of the Ni–Fe and the Mn–Ni layers. In order to make two different residual impurity levels in the sputtering atmosphere, the base pressure of the sputtering chamber was prepared as 5×10^{-11} Torr in the extremely clean (XC) process and 3×10^{-7} Torr for the lower grade (LG) process. By using a turbomolecular pump, we achieved the LG process within an hour after venting the chamber with air. After pumping the chamber for 30 h including 150 °C×24 h baking, the XC process was realized. A major residual gas in the LG process was H_2O, and only H_2 gas remained in the XC process. For the process gas, ultraclean Ar gas (UC-Ar), whose moisture level is less than 1 ppb, was used.[12] The flow rate of the UC-Ar gas during sputtering was 50 sccm to make the pressure ~1 mTorr. The *MH* loop was measured by a vibrating sample magnetometer. A unidirectional anisotropy constant J_k was calculated as $M_s d_F H_{ex}$, where H_{ex} is the exchange coupling field, determined as a shift of the center of the *MH* loop. The microstructure of the films was examined by x-ray diffraction (XRD) with a Co $K\alpha$ radiation source and a transmission electron microscope (TEM).

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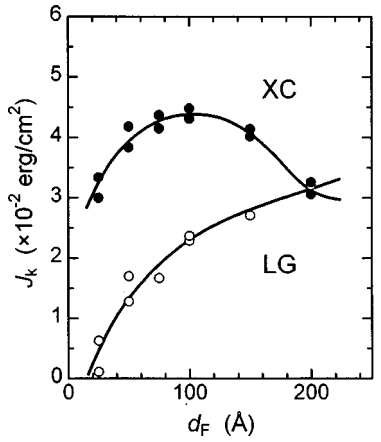

FIG. 1. Changes of unidirectional anisotropy constant J_k as a function of the ferromagnetic layer thickness d_F. The antiferromagnetic layer thickness d_{AF} was fixed at 300 Å.

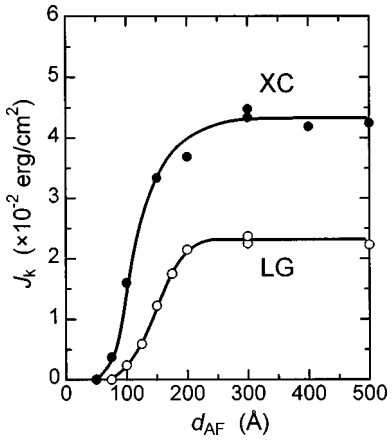

FIG. 2. Changes of unidirectional anisotropy constant J_k as a function of the antiferromagnetic layer thickness d_{AF}. The ferromagnetic layer thickness d_F was fixed at 100 Å.

III. RESULTS AND DISCUSSION

Figure 1 shows the changes of J_k as a function of the F layer thickness, when d_{AF} was fixed at 300 Å. In the LG processed films, J_k, which was about 0.005 erg/cm^2 at $d_F = 25$ Å, gradually increased with increasing d_F and became 0.03 erg/cm^2 at $d_F = 200$ Å. This increase of J_k in the very thin d_F region is caused by the increase of AF grain size.[13] In contrast, in the XC processed films, J_k was 0.03 erg/cm^2 even in the case of $d_F = 25$ Å and increased to a peak value of 0.045 erg/cm^2 at $d_F = 100$ Å. The enhancement of J_k of the XC processed films is correlated with the decrease of impurities in the films contaminated during the deposition process, because the fabrication conditions of both processed films are only different in the base pressure of the sputtering chamber. By secondary ion mass spectrometry, we have confirmed that the concentration of oxygen ($m/e = 16$), the major impurity element, in the XC processed film is lower than that in the LG processed film by one order of magnitude. Other impurity (N,C) concentrations were not different in both processes. We can build up two hypotheses about the effect of lowering the impurity concentration in the XC processed films on the exchange biasing. The first is a direct effect of impurities and easily imagined: (1) Impurities at the interface preventing the exchange coupling between F and AF atoms were cleaned out and the coupling energy per unit interface area J_k was enhanced. The second one is an indirect effect of the impurities: (2) The microstructure of the AF layer changed with the decreasing impurities. To put it clearly, the mean size of the AF grains increased with the decreasing impurities and the number of AF grains which contribute to the exchange biasing of the F layer at room temperature increased, because the distribution of the blocking temperature of the AF grains (the so-called ''local blocking temperature distribution'') became narrow and shifted to higher temperature with increasing AF grain size.[14] In order to examine these hypotheses, the critical thickness of the AF layer was investigated for both processed films. If there is no difference between the microstructure of the AF layers and the former hypothesis is correct, the critical thickness, defined as J_k/K_{AF} (Ref. 15) for the XC processed films should be greater than that for the LG processed ones. Here, K_{AF} is a magnetic anisotropy energy of the AF layer.

Figure 2 shows the changes of J_k as a function of the AF layer thickness, when d_F was fixed at 100 Å. In the LG processed films, J_k rose from $d_{AF} = 100$ Å and became constant at 0.023 erg/cm^2 when $d_{AF} > 200$ Å. On the other hand, J_k in the XC processed films rose from $d_{AF} = 75$ Å and became constant at 0.043 erg/cm^2 when $d_{AF} > 300$ Å. According to Mauri *et al.*, we can estimate the critical thicknesses of the AF layer, where J_k becomes half of the saturated values;[15] they are 110 and 150 Å for XC and LG processed films, respectively. This result means that the former hypothesis cannot be applied.

Figure 3 shows the changes in the XRD profile of the quadrilayer films for various d_F with $d_{AF} = 300$ Å. In both processed films, the diffraction intensity from the Ni–Fe (111) planes around $2\theta = 51.5°$ becomes gradually stronger with increasing d_F, while there is an obvious difference in intensity between the XC and the LG processed films. The most important differences that should be noticed among the XRD profiles of both processed films are the intensity and the width of the diffracted lines from the Mn–Ni (111) planes around $2\theta = 49.5°$. From these profiles, the sizes of the antiferromagnetic Mn–Ni grains along the film thickness were estimated by using Scherrer's formula.[16] Figure 4

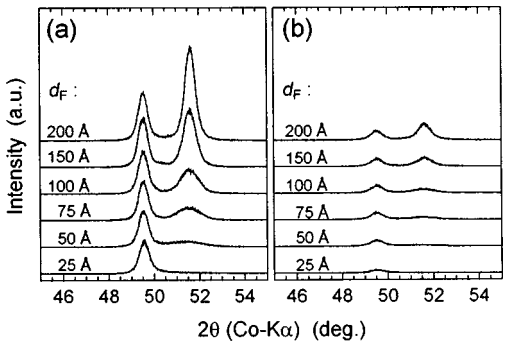

FIG. 3. Changes in XRD profiles of Ta 50 Å/Ni–Fe d_FMn–Ni 300 Å/Ni–Fe 1.5$\times d_F$ quadrilayer films fabricated under (a) the XC process and (b) the LG process.

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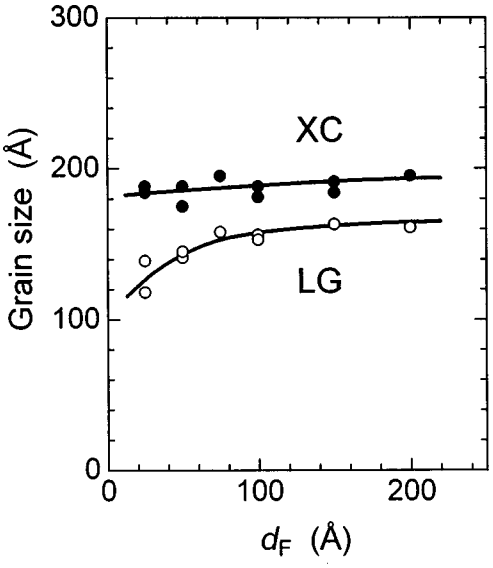

FIG. 4. Changes of antiferromagnetic grain size of Ta 50 Å/Ni–Fe d_F Mn–Ni 300 Å/Ni–Fe 1.5×d_F quadrilayer films as a function of d_F.

shows the changes of grain size of Mn–Ni as a function of d_F, when d_{AF} was fixed at 300 Å. The AF grain size of the XC processed films are larger than that of the LG processed ones. Especially on the very thin (~25 Å) F layer the difference is remarkable. The AF grain size of the XC processed films remains large (~180 Å), however, that of the LG processed ones becomes small (~120 Å). The decrease of the AF grain size results in the weakening of J_k at room temperature because of the thermal agitation of the AF spins.[14,17,18] In other words, the anisotropy energy ($K_{AF}\nu$) of the small AF grains are not necessarily enough to adhere the AF spins to the anisotropy axis unidirectionally when the F layer spins are reversed by an external field. Therefore, we can say that the large grain size of the antiferromagnet in the XC processed films shown in Fig. 4 is an origin of the enhancement of the exchange anisotropy in the very thin thickness region.

Figure 5 shows bright-field TEM images of 100 Å thick Ni–Fe layers fabricated on 50 Å thick Ta buffer layer under the XC and the LG processes, respectively. Electron diffraction patterns of both processed films showed fcc-[111]-incident ring patterns. In Fig. 5, we found that the in-plane diameters of the grains of the XC processed film are in some degree larger than that of the LG processed one, and that a high diffraction contrast of grains in the XC processed film. Namely, the fcc-[111] direction of the Ni–Fe grains in the XC processed films is highly oriented normal to the film plane. Since a Mn–Ni grain grows by epitaxy on a Ni–Fe grain, the preferred orientation of Mn–Ni grains traces that of the Ni–Fe grains. Taking into account that generally fcc (111) is a preferential growing plane, the AF grains in the

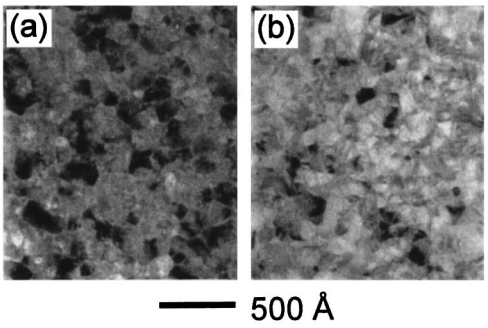

FIG. 5. TEM images of 100 Å thick Ni–Fe films fabricated on 50 Å thick Ta buffer layers under (a) the XC process and (b) the LG process, respectively.

XC processed films can grow perpendicularly to the film plane and become larger than that in the LG processed ones.

In summary, the exchange anisotropy of the Ni–Fe/Mn–Ni films fabricated under the different purities of the sputtering atmosphere were investigated in connection with the microstructure of the films. The enhanced exchange anisotropy in the XC processed films was found when the thickness of both ferromagnetic and antiferromagnetic layers were very thin. We conclude that the enhancement of the exchange anisotropy was caused by the enlargement of the antiferromagnetic grains in the XC processed films.

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