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Role of oxygen in the film growth and giant magnetoresistance of Co/Cu multilayers

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In order to clarify the effect of oxygen in the sputtering atmosphere on the microstructure and giant magnetoresistance, Co/Cu multilayers were fabricated under a sputtering atmosphere into which regulated impurity oxygen gas was introduced. After being pumped down the sputtering chamber to the ultimate pressure (less than 1×10^{-10} Torr), oxygen was introduced into the chamber until its content in processing Ar gas was about 0.1 ppm to 0.1%. The magnetoresistance (MR) ratio drastically increased from less than 20% to 54% when the content of impurity oxygen was slightly increased from 20 to 80 ppm, then nearly vanished when the content became more than 200 ppm. In the former region where the MR ratio steeply increased, the root mean square roughness of the multilayers decreased from 6.5 to 4.5 Å accompanied by a reduction in grain size as the oxygen content was increased. The partial oxidation of the multilayers is the most probable mechanism by which the flattening of the interfaces in the multilayer can be explained. We conclude that the impurity oxygen in the sputtering atmosphere serves as an obstruction of grain growth in the multilayer, not as a surfactant for the film growth.
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I. INTRODUCTION

The giant magnetoresistance (GMR) effect in metallic multilayers has been actively investigated by many researchers in the last decade.[1] In order to obtain a high magnetoresistance (MR) ratio, the general deposition conditions, such as deposition rate,[2] processing gas pressure,[3–5] substrate bias[4]/temperature,[6] etc., have been varied in order to control the microstructure of the multilayers. However, the most important factors concerning the general deposition conditions by which a high MR ratio can be obtained vary according to different reports and cannot be uniquely specified. This chaotic situation seems to be caused by the fact that it is not known which parameters must be controlled in order to realize a desirable microstructure and interface of metallic multilayers.

The purity of the sputtering atmosphere is believed to be one of those parameters because it is widely known that the impurities in the sputtering atmosphere strongly affect the magnetic properties accompanied by a significant change in the microstructure of magnetic thin films.[7–10] For Co/Cu multilayers, changes in the MR ratio against the base pressure of the sputtering chamber have been investigated by a few researchers.[2,11–13] In those articles, the MR ratio of the multilayer, whose Cu thickness (~9 Å) corresponds to the first antiferromagnetic (AF) coupling region, monotonously increases accompanied by a decrease of impurities in the thin film as the base pressure of the sputtering chamber is lowered down to about 4×10^{-8} Torr. Furthermore, this increase of MR ratio is attributed to the increase in the amount of antiparallel alignment of the magnetization of neighboring Co layers at zero field, associated with the increase of AF coupling energy. These results are naturally acceptable because the AF coupling between Co layers is introduced by the conduction electrons, which are diffusively scattered by the impurities in the film. However, our experimental results were not easily explained as a simple extension of the earlier results mentioned above. In particular, a significant decrease in the MR ratio was found when the base pressure was lowered from 7×10^{-8} to 1×10^{-10} Torr by changing the pumping time after venting the chamber with air.[14] In this case, the maximum resistivity change, $\Delta\rho$, dropped drastically without any change of saturated resistivity. This result indicates that residual impurities in the sputtering chamber, taken into the multilayer, act not only as diffusive scattering centers for conduction electrons but are also factors affecting the magnetic properties accompanied by the change of microstructure in Co/Cu multilayers. Therefore, it is very important to specify the dominant species of impurities affecting GMR and to clarify their role in the microstructure of multilayers.

Oxygen is one of the most significant impurities in the sputtering atmosphere affecting the GMR of multilayers. However, oxygen is reported to have different roles according to different researchers, and it has not been fully understood. Egelhoff *et al.* investigated the influence of various impurity gases on GMR in spin valves, and the MR ratio in the spin valves was found to be enhanced by introducing small amounts of oxygen up to 10^{-9} Torr in a vacuum range into the sputtering chamber. Based on these data, they proposed that oxygen acts as a surfactant during film growth to improve the surface/interface structure and grain size of the spin valves; namely, (1) the surface/interface roughness is reduced and (2) the grain size is increased due to the existence of oxygen.[15] On the other hand, Takahashi *et al.* reported a steep decrease of the MR ratio in spin valves when the Co surface was exposed to an oxygen atmosphere more

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than 20 L and explained that the reason for this phenomenon was the nanoscaled oxide layer formation on the Co surface.[16]

In this study, in order to clarify the role of oxygen on GMR more precisely, Co/Cu multilayers were fabricated under different sputtering atmospheres in which the impurity oxygen content was changed dramatically from 0.1 ppm to 0.1% within processing Ar gas. The correlation between the MR ratio and microstructure of the multilayers, especially the interfacial roughness, was investigated as a function of the partial pressure of oxygen introduced into the sputtering chamber.

II. EXPERIMENTAL PROCEDURES

Multilayers, of the form substrate/(Co 10 Å/Cu d_{Cu})$_{30}$/Cu 20 Å, were deposited on thermally oxidized Si(100) wafers at room temperature using a dc magnetron sputtering machine which was capable of pumping gases down to 4×10^{-11} Torr. The Cu layer thickness, d_{Cu} was optimized to make the MR ratio maximum in the so-called ''first peak'' of the GMR oscillation which ranged from 8 to 11 Å. After being pumped down to the ultimate pressure, *oxygen* was introduced through a variable leak valve to vary the partial pressure of the chamber from 10^{-10} to 1×10^{-6} Torr. The ultraclean Ar gas (UC-Ar), whose impurity level is less than 1 ppb,[7] was then introduced to make the total chamber pressure reach 0.6 mTorr. For the sake of comparison, *moisture* and *nitrogen* were independently introduced as impurity gases instead of *oxygen* for the same experimental purpose. The microstructure of the multilayers was analyzed by x-ray diffraction (XRD), x-ray reflectivity with a Cu $K\alpha$ radiation source, and atomic force microscopy (AFM). *M–H* loops were measured by a vibrating sample magnetometer (VSM) at room temperature. The oxygen content in Co/Cu multilayers was estimated by secondary ion mass spectroscopy (SIMS). The magnetoresistance was measured by a dc four-point probe method in a magnetic field up to 13 kOe at room temperature. The MR ratio was defined as $\Delta\rho/\rho_{13\,\mathrm{kOe}} \equiv (\rho_0-\rho_{13\,\mathrm{kOe}})/\rho_{13\,\mathrm{kOe}}$, where ρ_0 is the maximum resistivity at around zero field and $\rho_{13\,\mathrm{kOe}}$ is the resistivity under the applied field of 13 kOe. The saturation field, H_s, was defined as the magnetic field where the resistivity is 1% larger than $\rho_{13\,\mathrm{kOe}}$ in the magnetoresistance curves, in which the contribution of the force effect in the high magnetic field is corrected, as shown in Fig. 1. An AF coupling energy, J, between adjacent Co layers in the multilayer was calculated as $M_sH_sd_{Co}/4$, where M_s and d_{Co} are the saturation magnetization and thickness of one Co layer, respectively.

III. RESULTS AND DISCUSSION

A. GMR and magnetic properties

Figure 2 shows the MR ratio and $\rho_{13\,\mathrm{kOe}}$ of multilayers as a function of the partial pressure of impurities. Here d_{Cu} was optimized at 9 or 10 Å to derive the maximum MR ratio. A drastic change in the MR ratio was seen around the 10^{-8}–10^{-7} Torr range of the partial pressure. Namely, the MR ratio, which initially has a low percentage without the introduction of any impurities, started to increase with an increase in the partial pressure of oxygen (P_{O_2}) from 1×10^{-8} Torr, reached a sharp peak of 54% around $P_{O_2}=5\times10^{-8}$ Torr, then immediately vanished. On the other hand, $\rho_{13\,\mathrm{kOe}}$ was almost constant at about 22 $\mu\Omega$ cm over the range of the partial pressure up to 1×10^{-7} Torr, meaning that the drastic change in the MR ratio was dominated by the intrinsic change in magnetoresistance, $\Delta\rho$. With a further increase in P_{O_2}, $\rho_{13\,\mathrm{kOe}}$ steeply increased. This increase corresponded to the oxidization of the multilayer, resulting in the decrease of the MR ratio. In the case of *moisture* as the impurity element, the change of the MR ratio against the partial pressure was similar to that in the case of *oxygen*, except for the magnitude of the maximum MR ratio and the partial pressure where the MR ratio reached its maximum. In contrast, in the case of *nitrogen*, the MR ratio did not show a remarkable change against the partial pressure ranging from 1×10^{-9}–1×10^{-6} Torr. From these results, one can find a drastic change in the MR ratio in the multilayers fabricated with *oxygen* or *moisture* as an impurity element introduced into the sputtering chamber. By taking into account the fact that the critical partial pressure of *oxygen* at which the MR ratio reached its maximum was almost half that of *moisture*, one can safely say that oxygen atoms (ions or radicals) are the dominant species affecting the appearance of GMR in

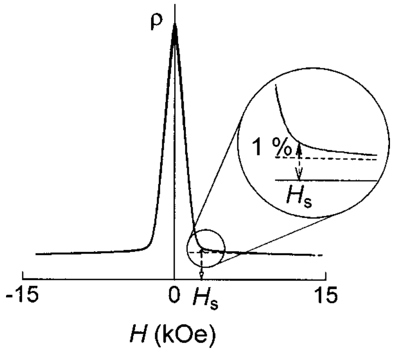

FIG. 1. Definition of H_s determined from a MR curve in the multilayer.

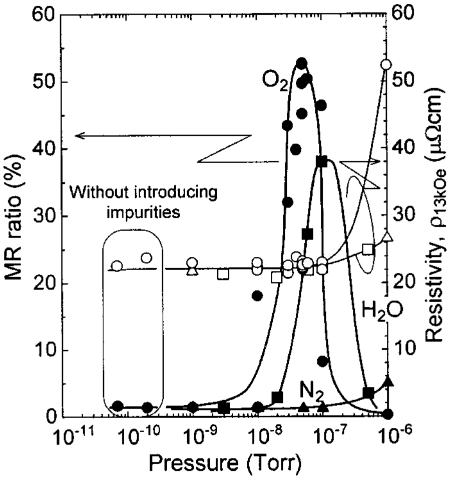

FIG. 2. Changes of MR ratio (solid marks) and $\rho_{13\,\mathrm{kOe}}$ (open marks) as a function of the partial pressure of oxygen (circles), moisture (squares), and nitrogen (triangles) introduced into the sputtering chamber.

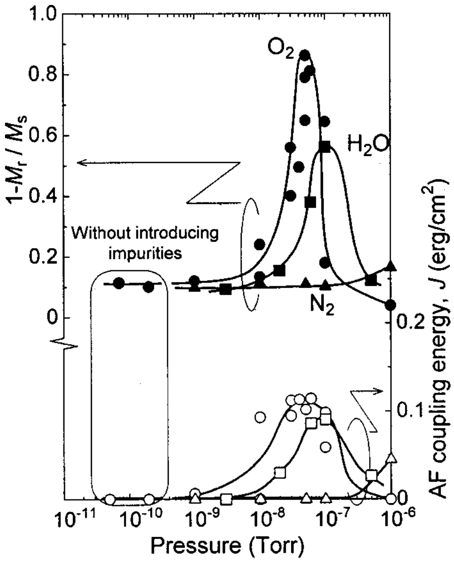

FIG. 3. Changes of $1-M_r/M_s$ (solid marks) and J (open marks) as a function of the partial pressure of oxygen (circles), moisture (squares), and nitrogen (triangles) introduced into the sputtering chamber.

metallic multilayers. It is noted that the optimum partial pressure of oxygen or moisture to obtain maximum MR ratio will depend on deposition systems. During deposition of multilayers, any surfaces that are coated with fresh films having high affinity for oxygen will help to pump the impurities. Since the partial pressure is measured before the deposition begins, the actual pressure of oxygen or moisture during deposition will be less. This drop in the impurity pressure will certainly be different in different deposition systems, depending on the area of the fresh film coverage and the rate of deposition.

In Fig. 3, $1-M_r/M_s$, the measure of the volume fraction of AF coupled regions of the Co layers at zero field, is plotted as a function of the partial pressure of impurity gases. The AF coupling energy, J, is also shown in the same figure. The remanent magnetization ratio, M_r/M_s was determined from an M–H loop measured along the easy magnetization axis of the multilayers. In the case of *oxygen*, $1-M_r/M_s$, which was about 0.1 without the introduction of any impurities, abruptly increased when P_{O_2} increased from 1×10^{-8} Torr, and made a sharp peak of 0.86 around $P_{O_2}=5\times10^{-8}$ Torr, then dropped to 0.2 at $P_{O_2}=1\times10^{-7}$ Torr.

J, which was almost zero in the multilayer fabricated without the introduction of any impurities, also increased with an increase in the partial pressure of *oxygen* from about 1×10^{-8} Torr, and dropped when the partial pressure of oxygen reached a point beyond 1×10^{-7} Torr.

These changes of two different kinds of physical quantities in relation to the partial pressure correspond well with the change of MR ratio shown in Fig. 2. In the case of *moisture*, the changes of both physical quantities in relation to the partial pressure were similar to those in the case of *oxygen*, and also correspond well to the change of MR ratio shown in Fig. 2. On the contrary, in the case of *nitrogen*, almost no effect was found within the pressure range examined.

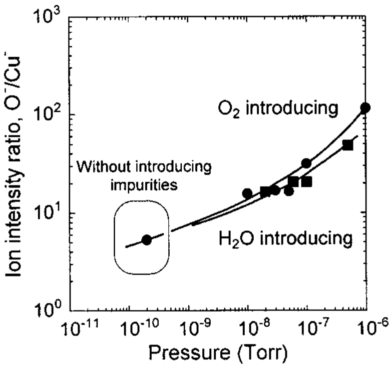

FIG. 4. Amount of oxygen in the multilayers as a function of the partial pressure of oxygen (circles) and moisture (squares) introduced into the sputtering chamber. The amount of oxygen was estimated as the ratio of oxygen ion intensity to copper ion intensity measured with SIMS.

From these results, one can see that the drastic change of MR ratio in the multilayers fabricated with *oxygen* or *moisture* introduced as impurities is derived from the change in the amount of antiparallel alignment of the magnetization of neighboring Co layers at zero field, which is associated with the change in AF coupling energy, J. Since the change in J is strongly connected to the changes in the microstructure of multilayers, in the following sections of this article we will discuss the microstructural factors which result in the diffusive scattering of the conduction electrons and the interfacial roughness in the multilayers.

B. Diffusive scattering centers for conduction electrons in the multilayers

In order to clarify the origin of the change in J in relation to the partial pressure of impurities, the amount of oxygen which was contained in the multilayers and induced microstructural changes, such as grain boundaries and defects, was examined.

Figure 4 shows the amount of oxygen in the multilayers as a function of the partial pressure of *oxygen* or *moisture* introduced into the chamber as an impurity. The amount of oxygen in the multilayers was estimated as the ratio of oxygen ion intensity (collected counts) to copper ion intensity measured with SIMS. The absolute value of the ion intensity ratio, O$^-$/Cu$^-$, does not show an actual content ratio in the multilayers because the relative sensitivities of SIMS were not calibrated for each element. Only the change in the ion intensity ratio against the partial pressure of impurities is significant and indicates the amount of oxygen atoms in the multilayers. In the case of the introduction of *oxygen*, O$^-$/Cu$^-$ monotonously increased with an increase in the partial pressure up to 1×10^{-6} Torr and became 10 times larger than that in the multilayer into which no impurities were introduced. In the case in which *moisture* was introduced, O$^-$/Cu$^-$ also increased monotonously in relation to the partial pressure, although oxygen atoms were less trapped inside the films in comparison with the case in which *oxygen* was introduced at the same partial pressure.

Figure 5 shows the changes in the XRD profile in the middle diffraction angle region of the multilayers, with d_{Cu}

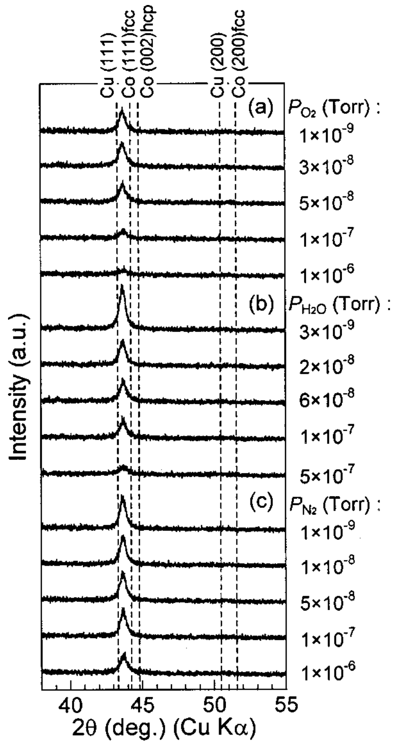

FIG. 5. Changes of x-ray diffraction profile in middle angle region of the multilayers fabricated in the sputtering atmosphere into which (a) oxygen, (b) moisture, and (c) nitrogen were introduced.

of 10 Å, fabricated under various partial pressures of (a) *oxygen*, (b) *moisture*, and (c) *nitrogen*. Interference peaks diffracted from both Cu (111) and Co (111) planes are observed around $2\theta=43.7°$ for all the multilayers. In the case of *oxygen* and *moisture*, the intensity of the interference peak became small with an increase in the partial pressure. At the same time, diffraction peaks from crystallographic planes other than (111) were not detected. These experimental results indicate the reduction of grain size and/or an increase in defects of grains in the multilayer with an increase in the partial pressure. On the other hand, in the case of *nitrogen*, the intensity of the interference peak almost did not change with an increase in the partial pressure. Taking into account the results shown in Fig. 4, one can say that oxygen atoms taken into the multilayers suppress the grain growth and/or induce defects of grains in the multilayer. The grain boundaries, which are due to the size reduction of grains, the defects of grains and the impurities trapped in the multilayer, are the diffusive scattering centers for conduction electrons, which mediate the interlayer coupling between Co layers. We thus conclude that the decrease of J when the partial pressure of *oxygen* or *moisture* is more than 5×10^{-8} Torr in this case (Fig. 3) is induced by an increase of such diffusive scattering centers for conduction electrons in the multilayers as previous researchers have discussed.[11,13] However, the decrease of J with a decrease in the partial pressure of *oxygen* or *moisture* below 5×10^{-8} Torr as shown in Fig. 3 cannot be explained by this mechanism.

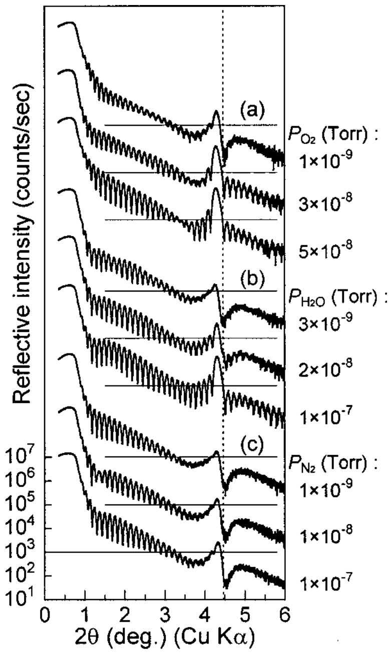

FIG. 6. Changes of x-ray reflectivity profile of the multilayers fabricated in the sputtering atmosphere into which (a) oxygen, (b) moisture, and (c) nitrogen were introduced. Horizontal lines are the guiding scale for comparison of the profiles and indicate the same reflective intensity of 10^3 counts/s for each profile.

C. Interfacial roughness in the multilayers

It is widely known that the interfacial roughness of multilayers induces a ferromagnetic (F) coupling between the adjacent magnetic layers through a nonmagnetic (Cu) layer: the so-called ‘‘orange peel effect.’’[17] According to this model, the F-coupling energy increases in a way similar to that of a quadratic function with a heightening amplitude of the sinusoidal waving interface, and this results in the decrease of the AF-coupling energy, J. One can thus expect the significant increase of the interfacial roughness of the present multilayers to explain the decrease of J with a decrease in the partial pressure of oxygen below 5×10^{-8} Torr.

Figure 6 shows the changes of the x-ray reflectivity profile of the multilayers, with d_{Cu} of 10 Å, fabricated under various partial pressures of (a) *oxygen*, (b) *moisture*, and (c) *nitrogen*. As a guiding scale for comparison of the profiles, 10^3 counts/s of reflective intensity are shown on each profile. The diffraction peaks originating from the artificial period are observed around $2\theta = 4.3°$. The observed peak position of these multilayers agrees with the one expected from the reflectivity calculation (a vertical dashed line in the figure) within 1-Å-thick deviation of the artificial period. In the cases of *oxygen* and *moisture*, the diffraction peaks and finite-size peaks, which appear as high frequency oscillations on the profiles, became large and clear with an increase in the partial pressure, indicating that the interfacial flatness in the multilayers improves with an increase in the partial pressure of *oxygen* or *moisture*. On the other hand, in the case of *nitrogen*, x-ray reflectivity profiles changed very little with an increase in the partial pressure up to 1×10^{-7} Torr.

To examine quantitatively the interface roughness in the multilayers, the root mean square (rms) roughness, σ, of

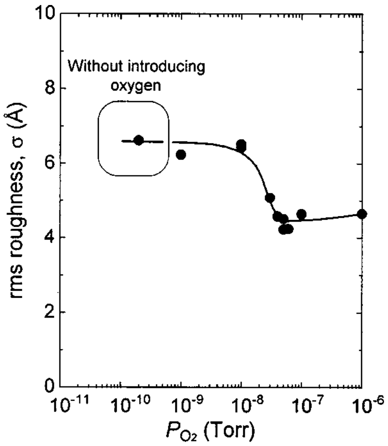

FIG. 7. Change of root mean square (rms) roughness as a function of the partial pressure of oxygen, P_{O_2}.

multilayers was estimated. In this study, the σ was calculated from the ratio of the observed reflectivity to the one calculated in the ideal multilayer with no roughness.[18] Figure 7 shows the rms roughness, σ, of multilayers fabricated with an introduction of *oxygen* as a function of the partial pressure of *oxygen*. The σ decreased from about 6.5 to 4.5 Å around $P_{O_2}=1\times10^{-8}$–5×10^{-8} Torr with increasing P_{O_2}. This change corresponds well to the steep increase of J shown in Fig. 3 and confirms that the multilayers with a large J should have flat interfaces.

The above results concerning the interfacial roughness in the multilayers can be verified by direct observation using an AFM. Figure 8 shows AFM images of the top surface of the multilayers fabricated under various P_{O_2}. The average surface roughness, R_a, decreased from about 4 to 2 Å with increasing P_{O_2} as well as the rms roughness in Fig. 7. This result supports that oxygen makes the films growth flatter as mentioned in Ref. 15.

Moreover, one can see the reduction in the lateral grain size of the multilayers with increasing P_{O_2}, which agrees well with the XRD results discussed in Sec. III B. This means that the decrease of interfacial roughness in the multilayers with an increase in P_{O_2} is caused by the suppression of the grain growth.

From these results, we conclude that the decrease of J with a decrease in the partial pressure of *oxygen* or *moisture* less than 1×10^{-7} Torr, as shown in Fig. 3, is caused by the deterioration of the flatness of the stacking structure in the multilayers, which enhances the *ferromagnetic* coupling due to the ''orange peel effect.''

D. Role of oxygen in the film growth

Figure 9 shows the changes in the MR ratio as a function of the number of bilayer stacks, N, in Co/Cu multilayers consisting of Si/SiO$_2$/(Co 10 Å/Cu 10 Å)$_N$/Cu 20 Å. The MR ratio increased monotonously with an increase in N when the partial pressure of impurity *oxygen* reached $P_{O_2}=5\times10^{-8}$ Torr. On the other hand, in the multilayers fabricated without introducing *oxygen*, the small percentage of the MR ratio was nearly independent of N.

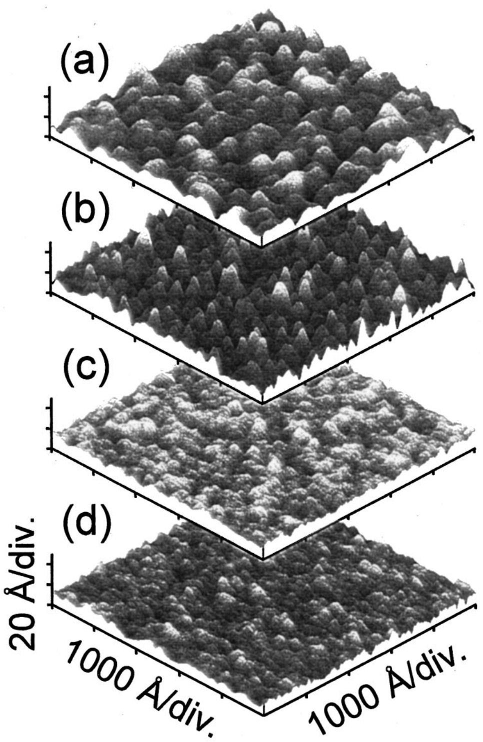

FIG. 8. AFM images of the top surface in the multilayers fabricated under various partial pressures of oxygen, P_{O_2}; (a) without introducing oxygen; (b) $P_{O_2}=1\times10^{-8}$ Torr; (c) $P_{O_2}=5\times10^{-8}$ Torr; (d) $P_{O_2}=1\times10^{-7}$ Torr. The average roughness, R_a, was (a) 3.4, (b) 3.9, (c) 1.9, and (d) 1.9 Å, respectively.

In order to clarify the role of oxygen, we examined a modified fabrication process for an $N=30$ bilayer as follows: the first ten bilayers in the multilayer were fabricated with an introduction of *oxygen* ($P_{O_2}=5\times10^{-8}$ Torr) and the subsequent 20 bilayers were fabricated without introducing *oxygen*. If the oxygen in the sputtering atmosphere acts as the so-called *surfactant*, which continuously segregates to the film surface and flattens the interface, the MR ratio of the modified multilayer should have nearly the same value as

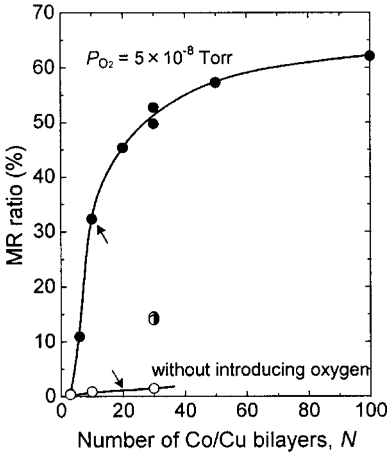

FIG. 9. Changes of MR ratio in the multilayers fabricated without oxygen (open circles) or with oxygen ($P_{O_2}=5\times10^{-8}$ Torr, solid circles) as a function of the number of Co/Cu bilayers, N. In addition, the MR ratio obtained in the multilayer fabricated under the modified process (half solid circles).

that of the multilayers with $N=30$ fabricated under $P_{O_2}=5\times10^{-8}$ Torr. However, the MR ratio observed in the modified multilayer was unexpectedly only about 15% (half solid circles in Fig. 9). To roughly assume the modified multilayer as a parallel resistance circuit consisting of ten bilayers fabricated under $P_{O_2}=5\times10^{-8}$ Torr and 20 bilayers fabricated without introducing *oxygen*, we can calculate the MR ratio by using 32% for the former and a small % for the latter (indicated by small arrows in Fig. 9). The calculated result gave an MR ratio of 15%. The remarkably good agreement of the calculated MR ratio with the experimental one indicates that the upper 20 bilayers in the modified multilayer have rough interfaces and show the same MR property as the 20 bilayers fabricated without introducing *oxygen* directly on the substrate. This means that oxygen does not act as the surfactant which continuously segregates to the film surface. Even if the oxygen has surfactant effect, the decay length of it to float out to the surface might be only a few nanometers as Ref. 15 suggests. However, judging from the reduction of the lateral grain size with increasing P_{O_2} shown in Fig. 8, we conclude that the main role of oxygen in the sputtering atmosphere is as an obstruction of grain growth in the multilayer. The partial oxidation of the multilayers is the most probable mechanism by which the reduced grain size and the flattening of the interfaces in the multilayer fabricated under $P_{O_2}=5\times10^{-8}$ Torr can be explained.

IV. SUMMARY

Co/Cu multilayers were fabricated in the sputtering atmosphere into which *oxygen* was introduced. The change of MR ratio in relation to the partial pressure of *oxygen* was investigated in connection with the microstructure in the multilayers. As a result of our investigations, we found that: (1) oxygen in the sputtering atmosphere strongly affected the GMR effect, and the MR ratio drastically increased from a low percentage to 54% with an increase in P_{O_2} up to 5×10^{-8} Torr; (2) the MR ratio abruptly decreased with an increase in P_{O_2} more than 1×10^{-7} Torr; (3) around $P_{O_2}=5\times10^{-8}$ Torr, the rms roughness in the multilayers decreased from 6.5 Å to 4.5 Å associated with the reduction of the grain size as P_{O_2} was increased; (4) the steep increase of the MR ratio was accounted for by the increase of J accompanied by the decrease of interfacial roughness; (5) the decrease of the MR ratio in the P_{O_2} more than 1×10^{-7} Torr was accounted for by the decrease of J accompanied by the increase of diffusive scattering centers in the multilayers; (6) the MR ratio in the modified multilayer which consisted of 10 Co/Cu bilayers fabricated under $P_{O_2}=5\times10^{-8}$ Torr and 20 bilayers fabricated without introducing *oxygen* was only about 15%.

We conclude that oxygen in the sputtering atmosphere acts as an obstruction of grain growth in the multilayer which changes the microstructure of the multilayer and that it is the most important process parameter to be controlled in order to obtain a high MR ratio.

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