Magnetotransport Studies in Co/Cu and Ag Nano-structure Films Prepared by Pulse Electrochemical Deposition

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The magnetotransport properties in Co/Cu, Ag multilayer films with layers produced in the atomic level by pulse electrodeposition method have been investigated. Magnetoresistivity, $\Delta\rho/\rho$ increases with increasing Co layer thickness and reaches a maximum in the neighborhood of 10 ~15 Å and 15 ~ 20 Å for the electrodeposited Co/Cu and Co/Ag multilayers, respectively. On the application of strain, these electrodeposited films show useful magnetic anisotropy properties. The magnetoresistance effect has two components: isotropic and anisotropic. The disparity in the magnetotransport properties seems to arise from the magnetic orientation in the ferromagnetic layer on both the isotropic and anisotropic films.

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Introduction

The research on the physical properties of the metallic multilayer structures prepared in an atomic scale has drawn a great deal of attention not only in fundamental physics but also in applications to electronic engineering [1-4]. Magnetoresistance effect [5] and anisotropy properties, which mainly carry significant interest in the data storage industries is interpreted by considering spin dependent scattering of conduction electrons [6-10]. On the other hand, magnetoresistance effect due to spin-orbit interaction in the ferromagnetic materials has long been known as anisotropic magnetoresistance (AMR) effect [11-12]. However, the relationship between the preparation conditions and the physical properties is not clear yet. We have previously reported [13] that electrodeposited Co/Au(111) structures exhibit magnetoresistance and magnetic anisotropy properties with almost equal strength to that of Co/Au multilayer grown by electron beam evaporation method [14].

Pulse electroplating is one of the useful techniques for the fabrication of metallic multilayer structures. It is possible to regulate the layer composition, its thickness, and the grain size in an atomic scale by normalizing the pulse amplitude and width. Electrodeposited layer structure has a possibility of forming a fine particle or pure layer depending on the preparation parameters, which has noteworthy consequence on the magnetic properties virtually different from those of the films fabricated by high vacuum evaporation techniques.

In this paper is discussed the preparation of nano-order scale Co/Cu, Ag multilayer films on polyimide substrates using a pulse generator circuit controlled by a microcomputer. The effect of magnetoresistance effect due to the spin arrangement in the adjacent ferromagnetic layers in both the multi domain and single domain multilayers is also studied. The effect of ferromagnetic Co layer thickness on the magnetoresistance and strain-induced magnetic anisotropy behaviors are examined by means of four-probe resistance measurement and vibrating sample magnetometer (VSM).

Experimental

The electrolytic bath for the Co/Cu multilayer film deposition was composed of $CoSO_4 \cdot 7H_2O$ $CuSO_4 \cdot 2H_2O$, $Na_3C_6H_5O_7$, NaCl, for Co/Ag deposition, $CoSO_4 \cdot 7H_2O$, AgI, KI, and that for Co/Au multilayer film $CoSO_4$ $7H_2O$, $KAu(CN)_2$, $Na_3C_6H_5O_7 \cdot 2H_2O$, NaCl. The solutions were prepared with pure chemicals in double distilled water, and the pH was adjusted in the range of 3.0 ~ 5.0. Preceding to the electrochemical fabrication of multilayer structures, substrates each 150 Å cupper buffer layer on polyamide materials was vacuum deposited unlike our previous reports [13, 15] in which multilayers were prepared using Cu buffer layers on glass and aluminum plates.

The deposition current efficiency of the ferromagnetic single layer is given below:

$$\text{Efficiency } (\eta) = \eta_{Co} + \eta_{Cu} = W_{Co.}\, \Delta G/E_{Co}/I.t + w_{Cu}\, .\, \Delta G/E_{Cu}/I.t \qquad (1)$$

where, η_{Co} and η_{Cu} are partial current efficiency, and k_{Co} (0.30539×10^{-3} g-C^{-1}) and k_{Cu} (0.3294×10^{-3} g- C^{-1}) are electrochemical equivalent values, ΔG is the amount of metal deposit in gram, w_{Co}

and w_{Cu} weight portion values of Co and Cu, respectively. Similarly, equation (1) was utilized for the preparation of Co/Ag (k_{Ag} =1.118 × 10^{-3} g-C^{-1}) multilayer structures.

A pulse generator circuit (as shown in Fig. 1) was designed and manufactured to fabricate multilayer and granular alloy structures on a thin copper buffer layer from an acidic sulfate solution with and without additives. The microcomputer was run by specially developed software to generate the pulse wave for electrochemical deposition. An output signal of the voltage regulated by the microcomputer is applied to the cathode electrode in the plating bath through the D-A converter and the constant current circuit.

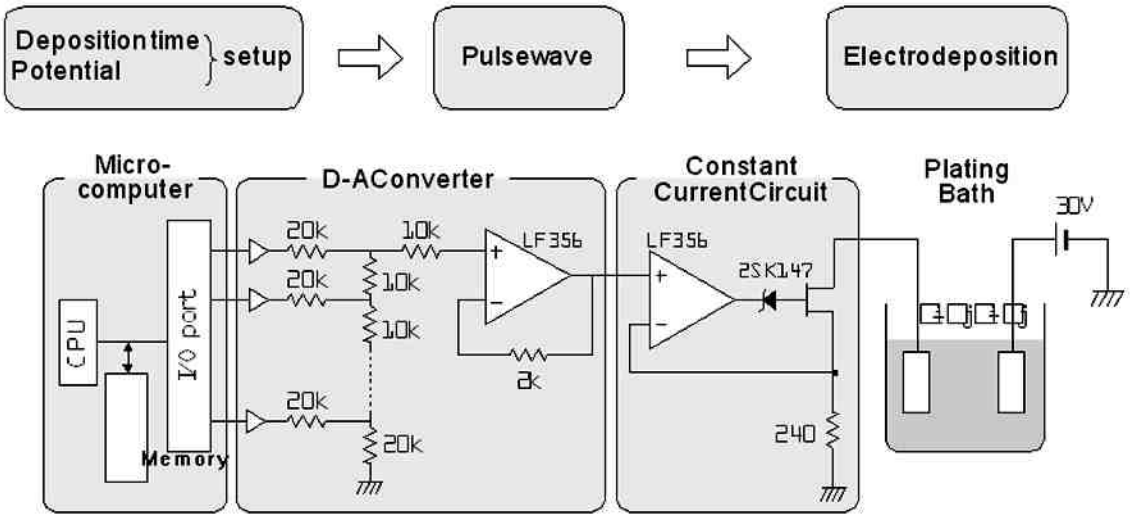

Fig. 1 shows the diagram of a pulse generator circuit. An output of the voltage regulator is applied to the cathode electrode in the plating bath through the D-A converter and the constant current source.

In the plating bath, Co concentration is changed while keeping the concentration of Cu and Ag constant; where as the ratio between the Co and Cu or Ag is varied. The amount of NaCl and Na citrate content of the bath is held constant, each to 5 g/L. For the Co/Cu multilayer, both Co to citrate ratio of the bath is changed with increasing Co concentration in the bath. The purpose of adding sodium chloride was to enhance the conductivity of the electrolyte. For the fabrication of Co/Ag multilayer films, no addition of NaCl was needed. The deposition was carried out at room temp. The use of Sodium citrate in the solution was to improve processing conditions and material properties. As the crystallization, kinetics, film structure and, therefore magnetotransport properties all depend on both the electrolyte composition (*e.g.* ion concentration, type and/ or amount of additive, pH) and plating parameters (e.g. current density, temperature, and convection), it was possible to produce Co/Cu and Co/Ag multilayer films having various composition and thicknesses by regulating pulse wave output from the constant current source.

Multilayer films were deposited using a square pulse wave of current density range 0.01-2 A/dm^2 for the Co/Cu multilayers and 0.01-2.5 A/dm^2 for the Co/Ag multilayer films. The ferromagnetic layer was Co-rich, with composition in Co/Cu and Co/Ag multilayer films was

90 at% Co-10 at% Cu and 98 at% Co-2 at% Ag, respectively. The multilayer parameters for the fabrication of Co-Au films is described elsewhere [18]. Various electrochemical parameters are summarized in the Table 1.

Table 1: Parameters for electrochemical deposition of nano-structure multilayer films

	Co/Ag	Co/Cu	Co/Au
Solution	$CoSO_4 \cdot 7H_2O$ AgI KI p.H. 3.0 Electrode: Ag	$CoSO_4.7H_2O$ $CuSO_4.2H_2O$ $Na_3C_6H_5O_7$ NaCl p.H. 5.0 Electrode: Cu	$CoSO_4.7H_2O$ KA(CN)2 NaCl $Na_3C_6H_5O_7$ p.H. 4.0 Electrode: Co
Current Density (J)	0.01~2.5 A/dm^2	0.03~2 A/dm^2	0.02-2 A/dm^2
Substrate	Polyamide, Cu	Polyamide, Cu	Cu, Al

A composition of the ferromagnetic and non-magnetic layers was determined by using atomic absorption spectroscopy, energy dispersive analysis, and highly sensitive quartz crystal microbalance. The experimental method using atomic absorption spectroscopy method involved two test runs in order to determine correctly the concentration of an element in the sample. At first, standard samples of known concentration (*i.e.* mg/ml) of the element under investigation were measured and their absorbance "A" is plotted against the concentration of the element. Secondly, the sample under investigation was dissolved in the acid e.g. HNO_3 or H_2SO_4 and subsequently diluted in a beaker containing the fixed amount of distilled or de-ionized water (20 ml beakers). The absorbance of the prepared solution of the sample is then measured with the spectrometer. By comparing the intensity of the absorbance of the sample with that of the standard one, the absolute concentration of both the metallic components (at %) were calculated. This was further verified by a highly sensitive quartz crystal microbalance by means of mass and thickness measurements. Magnetic properties were investigated by using a vibrating sample magnetometer (VSM). It has been reported [17] that electrodeposited Co/Ag films exhibit magnetic anisotropy behavior on impressing strain. This was further verified with Torque meter. A Strain gauge was used to measure the strain in the multilayer film deposited on the 150 Å Cu buffer layers on the polyamide film. The concentration of the metals as determined from the atomic analysis is then used to calculate magnetization from the following mathematical relation:

$$I = \frac{M_S}{t_{Co}} \text{ emu/Å} \qquad (4)$$

where, "M" is magnetization obtained from the field dependence of the magnetization curves, and "t" is the thickness of the ferromagnetic component Co.

Multilayers with cross-sectional area each with 1 cm^2 were fabricated for the magnetotransport studies in both the anisotropic and isotropic multilayer films. Measurements were carried out in four configurations as shown in Fig. 2, i.e. with the sensing current I and orientation of the easy and the hard axis parallel and perpendicular to the external field H. Fig

2(a) shows the magnetoresistance measurement for the isotropic film and (b) shows for the anisotropic films. Corresponding arrows show off the spin arrangement, easy axis and hard axis, direction of applied field, and current flow. Magnetic field dependence of MR ratio was examined by varying the relative direction of the magnetic field H and current I. MR ratio along the transverse and longitude direction of the measuring current was observed by measuring in the two configurations for the magneto-isotropic film. For the films with uniaxial anisotropy, MR ratio was measured in the four configurations by varying the relative direction of the field H and current I. MR ratio using a dc four-probe method was measured under the magnetic field range of 0 to 20 kOe. MR ratio was calculated using the following mathematical relation:

$$\%MR = \frac{R_H - R_0}{R_0} \times 100 \qquad (2)$$

where, R_H = Resistance of the sample at the applied magnetic field
R_0 = Resistance of the sample at zero applied field.

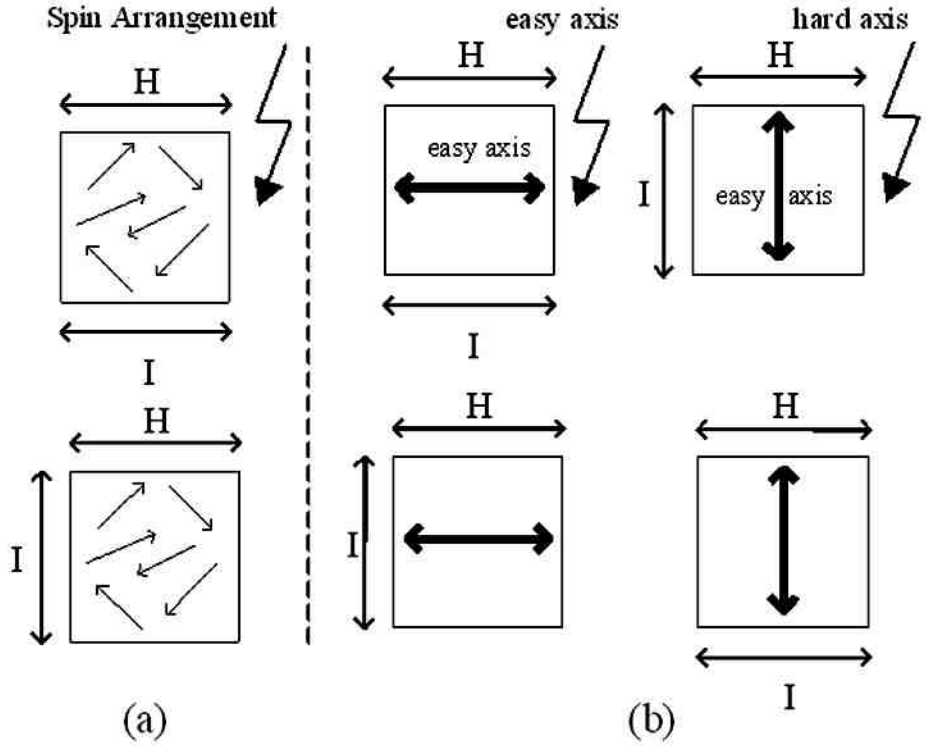

Fig. 2 Measurement of magnetoresistance in four configurations for the sensing current (I) and magnetic field (H) parallel and perpendicular to the orientation of the easy and hard axis; (i) I // $H_{e.a.}$, (ii) I $\perp$ $H_{e.a.}$ (iii) I // $H_{h.a.}$ and I $\perp$ $H_{h.a.}$ for the (a) isotropic and (b) anisotropic multilayer samples

The AMR ratio in ferromagnetic films was calculated as

$$\%\,\mathrm{AMR} \equiv \frac{(R_{//} - R_{\perp})}{R_0} \times 100 \qquad (3)$$

where, R_0 is the averaged electrical resistance, and $R_{//}$ and $R_{\perp}$ are the saturated longitudinal and transverse magnetoresistance, respectively. All the measurements were performed at 300 ^{0}K with in plane magnetic field.

Results and Discussions

*Fabrication of Multilayer Structure Using Pulse Electrochemical De*position

To prepare samples containing a range of composition of the ferromagnetic and non-magnetic particles that reveal large magnetoresistance effect, high sensitivity and magnetic anisotropy properties, it is crucial to vary the current density and examine the deposition pattern of the alloy film with respect to the bath. Moreover, it is also equally important to investigate the relationship between the Co content of the deposit and the metal concentration of Co in the bath for understanding the co-deposition of Co and Cu or Ag from the sulfate bath.

A multilayer film can be fabricated by means of electrochemical deposition from a single electrolyte[16] containing salts of two components (metals) of the film. In the present work, a single electrolyte containing ferromagnetic Co and non-magnetic Cu or Ag is employed to fabricate the multilayer structure. Fig. 3 shows the correlation of Co concentration between the electrolyte and the single layer deposited at the current densities of 0.03 and 2 A/dm^2 (Co/Cu: closed circles) and 0.01 and 2.5 A/dm^2 (Co/Ag: open circles), respectively. Ferromagnetic Co concentration of the deposited films is lower in the lesser concentration region of the bath. However, the concentration in the film remarkably increases for the bath composition in excess of 50 at% of Co. The composition of Co in the film tries to reach the composition of film at the higher current density *i.e.* current density of about 1A/dm^2 in our experiment.

The inset shows a multilayer deposition principle of the pulse electrochemical method. As it can be seen, almost a pure layer of the more noble metal (metal A: Cu or Ag) is deposited by choosing the potential (current density) to be between the reduction potential of the two metals, however, when an apposite value of reduction potential is selected for the deposition of the less noble metal, there is a likelihood of resulting an alloy (A + B: Co + Cu or Ag) layer rather than a pure layer. At potential V_1 (corresponding to the current density, J_A), a pure layer of Cu or Ag nonmagnetic metal is deposited. At potentials V_2 (corresponding to the current density J_{A+B}), Co-rich layer is deposited. By switching the potential between V_1 and V_2, and controlling the deposition time (width), multilayers of ferromagnetic Co-rich layer and non-magnetic Cu and Ag layers were fabricated. Similar tendencies were also observed for the Co/Au multilayer films reported elsewhere[15,18].

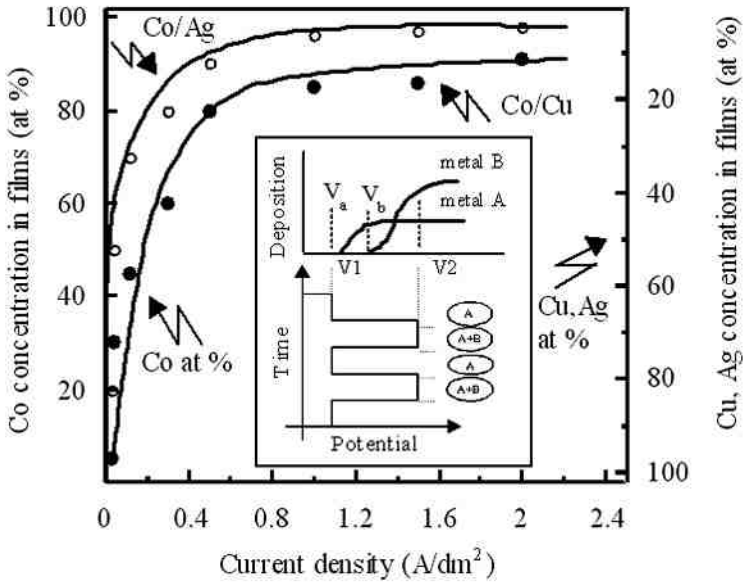

Fig. 3 Concentration of Co in electrodeposited films as a function of the deposition current density. o (Open circle): Co at% in the ferromagnetic layer of Co/Ag films, and • (closed circles): Co at% in the ferromagnetic layer of Co/Cu multilayer films

Magnetization curves and field sensitivity of MR ratio at low Field

Fig. 4 shows the magnetic field dependence of the MR ratio and corresponding magnetization curves for the randomly oriented [Co 10 Å/Cu 15 Å]$_{50}$ and [Co15 Å /Ag 15 Å]$_{30}$ multilayer films: (a) MR ratio of the Co/Cu randomly oriented film, and (b) MR ratio of the Co/Ag randomly oriented film. A remarkable strain induced magnetic anisotropy is observed which can be seen in the inset at the upper right corner. The field dependence of MR ratio for the anisotropic films also showed similar tendency. In the weak magnetic field ($\pm$ 1 kOe), the difference of magnetic field dependence of MR ratio is clearly observed with depending on shape of the magnetization curve. The evidences show the isotropic character of the MR ratio. As it can be seen, the measurement of MR-H curve shows an abrupt change of negative magnetoresistance, that is, they show high sensitivity at low magnetic field. As compared to the Co/Cu multilayers, the sensitivity of MR ratio to the external magnetic field for the Co/Ag multilayer is more significant.

The magnetic spin state near zero magnetization for the randomly oriented sample is antiparallel, where electric resistance shows high value. Therefore, the orientation of the magnetic spin for the isotropic films remarkably corresponds to the value of the MR ratio. The field dependence of MR ratio does not significantly depend on the direction of measuring current, but it only has a tendency to depend on the direction of the applied field.

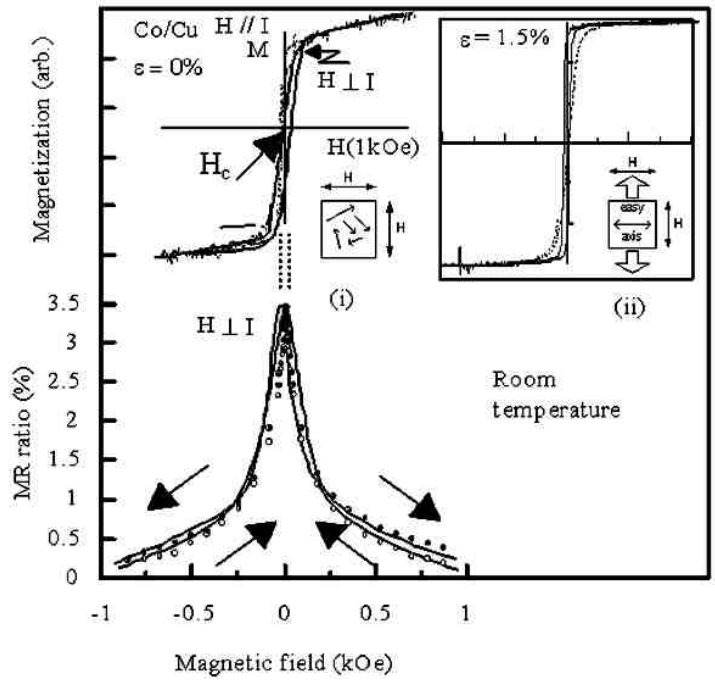

Fig. 4 (a) Magnetic field dependence of the MR ratio for the [Co 10 Å/Cu 15]$_{50}$ isotropic multilayer films and corresponding magnetization curves for (i) randomly oriented i.e. isotropic and (ii) anisotropic films

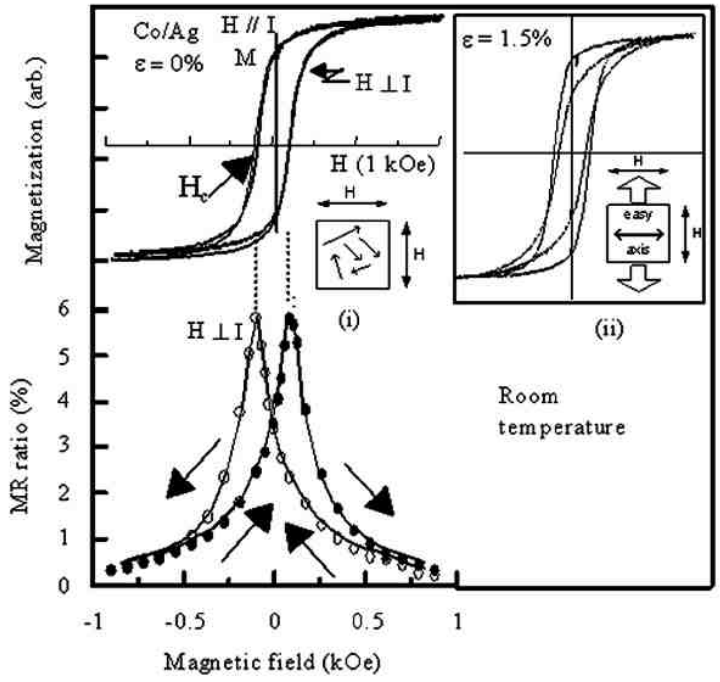

Fig. 4 (b Magnetic field dependence of the MR ratio for the [Co 15 Å/Ag 15 Å]$_{30}$ isotropic multilayer films and corresponding magnetization curves for (i) isotropic and (ii) anisotropic multilayer films.

Ferromagnetic layer thickness dependence of Magnetoresistance

The dependence of the absolute value of % MR ratio (H=20 kOe) on the ferromagnetic Co layer thickness, t_{Co} for constant non-magnetic layers of t_{Cu} and t_{Ag} is studied for both the isotropic and anisotropic multilayer films. The absolute value of MR ratio |MR% (H = 20 kOe)|, against Co layer thickness (t_{Co}) for t_{Cu} =10 Å and t_{Ag} = 15 Å is plotted for three set of samples i.e. isotropic and anisotropic multilayer films. Fig. 5 (a) shows ferromagnetic layer thickness dependence of MR ratio for the [Co t Å /Cu 10 Å]$_{50}$ multilayer:
(open square) indicates the MR ratio of the randomly oriented film, ● (closed circles), and ○ (open circles) are MR ratio of uniaxially oriented films, where (b) measuring current // magnetic easy direction (c) measuring current // magnetic hard direction. The ferromagnetic layer thickness (t_{Co}) is varied in the range of 5 ~ 20 Å for the Co/Cu multilayer system. Similarly, Fig. 5(b) shows the Co layer thickness dependence of the MR ratio for the Co/Ag multilayer with t_{Ag} = 15Å and Co layer thickness varied in the range of 5 ~ 30 Å. Solid lines are drawn as a direction for the eyes. It can be seen that these curves reach a clear broad maximum when varying Co layer thickness in each of the multilayer film.

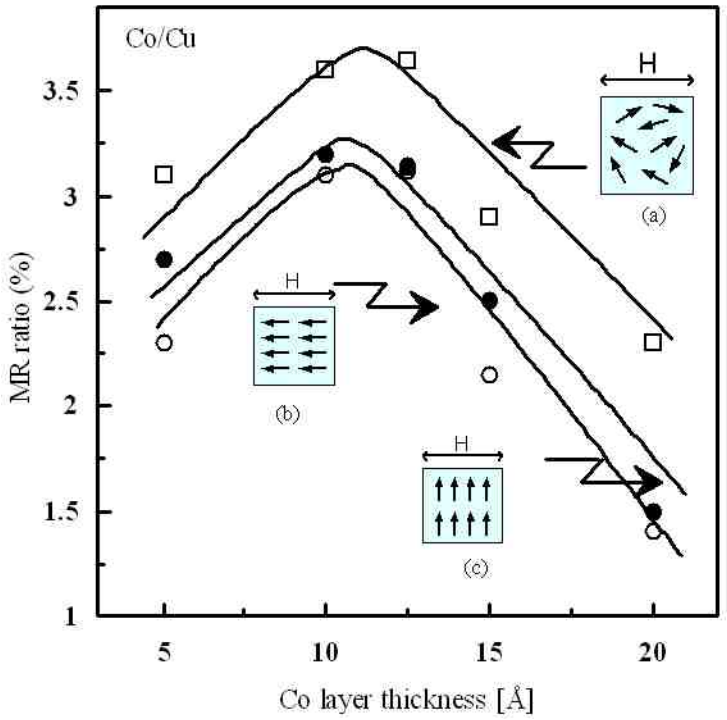

Fig. 5 (a) shows ferromagnetic layer thickness dependence of MR ratio for the [Co t/Cu 10 Å]$_{50}$ multilayer. (open square) indicates the MR ratio of the randomly oriented film, ● (closed circles), and ○ (open circles) are uniaxially oriented films, where (b) measuring current // magnetic easy direction (c) measuring current // magnetic hard direction

MR ratio reaches maximum of 3.7% for Co/Cu and 9.2 % for Co/Ag multilayer films. These values significantly decrease for higher t_{Co} when varying in each of the series for t_{Cu} 10 Å, and

t_{Ag} = 15 Å, respectively. The measuring directions are shown in Fig. 2. MR ratio of the isotropic sample shows the average of two values measured by changing the orientation of measuring current at the field direction of the applied field. Similarly, MR ratio of uniaxial anisotropy samples show the averages of the two values measured by changing the direction of measuring current at the fixed applied field along to the magnetic easy and hard axis.

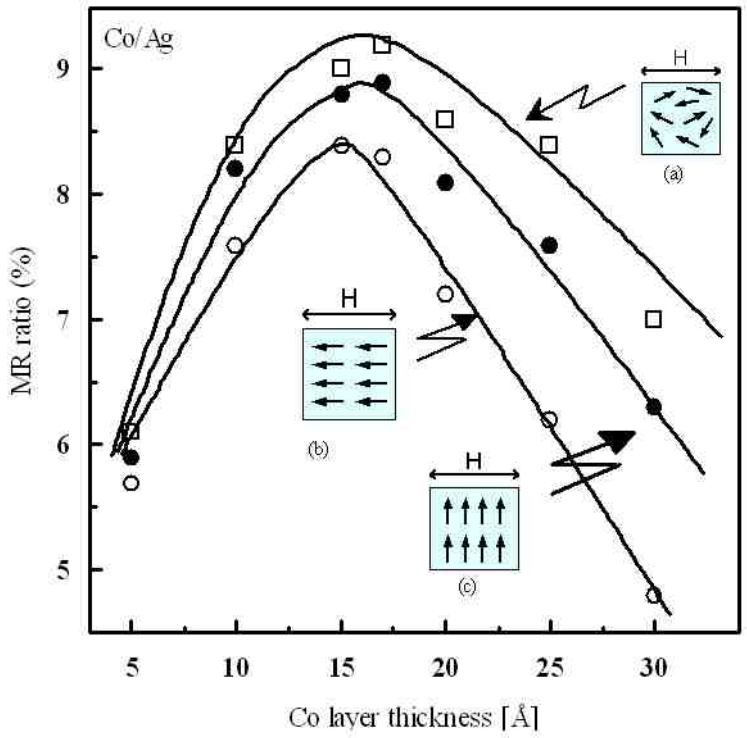

Fig. 5 (b) shows ferromagnetic layer thickness dependence of MR ratio for the Co/Ag multilayer. (open square) indicates the MR ratio of the randomly oriented film, ● (closed circles), and ○ (open circles) are uniaxially oriented films, where (b) measuring current // magnetic easy direction (c) measuring current // magnetic hard direction

These result suggest that the adequate thickness of Co ferromagnetic layer and Cu or Ag non-magnetic layers are required to create the high sensitivity and the large magnetoresistance ratio in Co-based multilayer films. MR ratio for the anisotropic multilayer films along the hard axis shows the value larger than that of the easy axis. The overall MR ratio of the isotropic film is larger than that of anisotropic films. The disparity in the magnetotransport properties seems to arise from the magnetic orientation in the ferromagnetic layer i.e. the number of antiparallel alignment of the magnetic spin in the alternate ferromagnetic layers adjacent to the non-magnetic Cu or Ag layer seem to be smaller than that of isotropic film. These results also suggest that an antiparallel alignment of magnetic spin is a necessary but not sufficient condition in order to generate the large magnetoresistance in the multilayer films.

Magnetic Field dependence of Magnetization Curves

Field dependence of magnetization curves for the series of strain-induced (ε =1.5%) Co/Cu and Co/Ag multilayers with varying t_{Co} were carried out to investigate magnetic anisotropy properties in the films. The relationships between β at room temperature, which is calculated from M_r(e.a.) and M_r(h.a.) and Co layer thickness for the [Co t Å /Cu 10Å]$_N$ and [Co t Å/Ag 15Å]$_N$ multilayers are shown in Fig. 6(a) and Fig. 6(b), respectively. Insets at the upper left side show the set of magnetization curves as a function of Co layer thickness. Magnetization curves were measured with magnetic field perpendicular (h.a.) and parallel (e.a.) to the measuring current direction measured by VSM. Magnetic field is increased from 1 kOe to +1 kOe and again completed to one full cycle. The magnetic anisotropy has rising tendency with the increase of ferro-magnetic layer thickness. The maximum of MR ratio in Fig. 5 is corresponding with the Co layer thickness of 10 Å (Co/Cu) and 15 Å (Co/Ag) in Fig. 6, at ε = 1.5%, respectively.

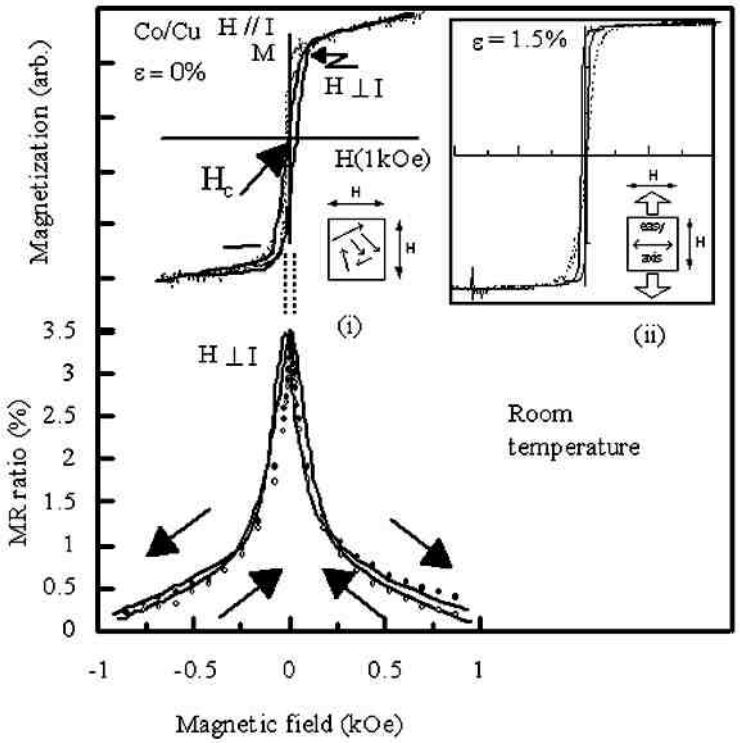

Fig. 6(a) Relationship between β and Co layer thicknesses in the range of 5~20 Å (ε =1.5%). Upper left inset shows the Co layer thickness dependence of the magnetization for the Co/Cu multilayer films at an applied magnetic field of 1kOe

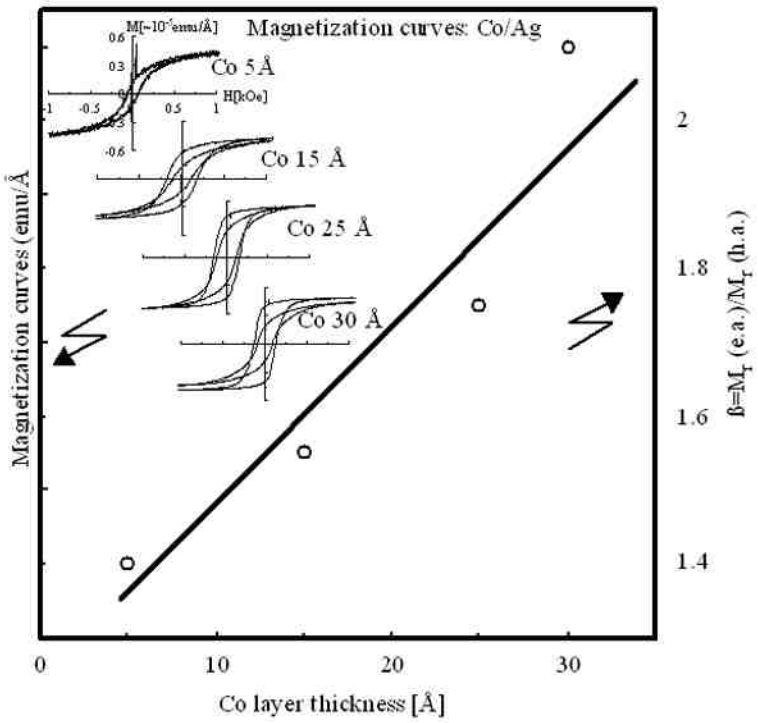

Fig. 6(b) Relationship between β and Co layer thicknesses in the range of t_{Co} 5~30 Å at an applied strain, ε of 1.5%. Inset shows the Ag layer thickness dependence of the magnetization curves for the Co/Ag multilayer films at an applied magnetic field of 1kOe.

Magnetic field dependece of MR ratio

Fig. 7 shows the field dependence of MR ratio for the Co/Cu multilayer films in the magnetic field range of ±20 kOe. The magnetoresistance effect has two components: isotropic and anisotropic. Result of magnet-isotropic film is given here. The difference in shape of MR-H curve was slighlty observed by changing the direction of applied field against the measuring current, which is shown in the upper side in Fig. 7. As the tendency of the field dependence in the lower side of the of Fig. 3 shows negative field dependence, this effect seems to be GMR effect. The MR ratio depends on the relative orientation of current and magnetic field which shows anisotropic character.

The MR-H curve shown in the upper side seems to arise from the anisotropic component of magnetoresistance effect. The isotropic component of MR ratio is attributed to the spin dependent scattering at the Co cluster surfaces. The disparity in the magnetotransport properties seems to arise from the magnetic orientation in the ferromagnetic layer.

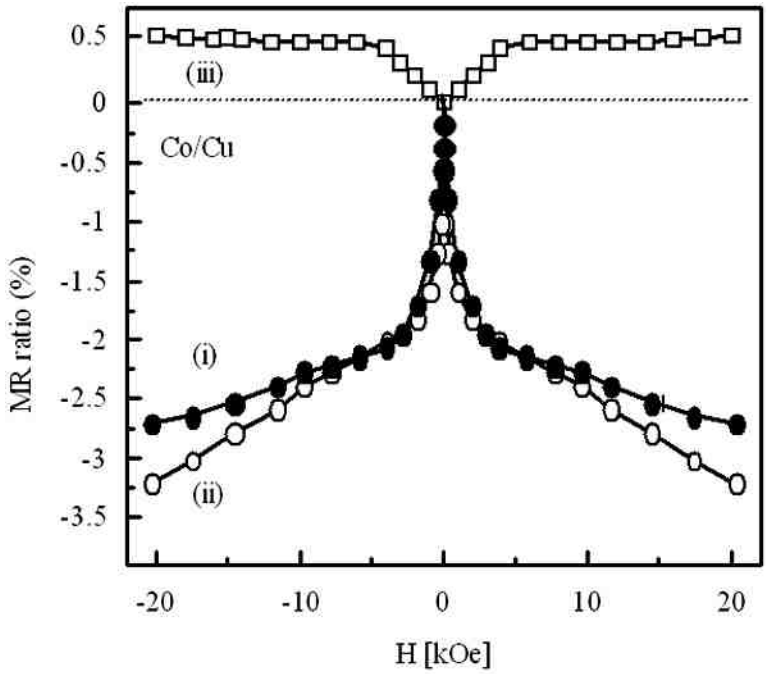

Fig. 7 Magnetic field dependence of the MR ratio for the [Co 10 Å/Cu 15]$_{50}$ isotropic multilayer films and for the randomly oriented films at the magnetic field of 20 kOe. Magnetic field (i) parallel (ii) perpendicular to the measuring current, and (iii) anisotropic magnetoresistance

Conclusion

It was possible to produce nano-order scale Co/Cu and Co/Ag multilayer films by pulse electrochemical deposition method. The relationship between the magnetoresistance and ferromagnetic layer thickness is examined. The magnetoresistance effect has isotropic and anisotropic components. The isotropic contribution in the as-deposited films arises from the formation of ferromagnetic Co multi-domain layers and the anisotropy contribution in the strain-induced films arises due to the formation of single domain cobalt particles at the interfaces between magnetic and nonmagnetic layers. Magnetoresistivity increases with increasing Co layer thickness and reaches a maximum in the neighborhood of 10~15Å and 15~20Å for the Co/Cu and Co/Ag multilayers, respectively. The difference in the magnetotransport properties seems to arise from the magnetic orientation in the ferromagnetic layer.

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