Magnetotransport Properties of Co-Au Granular Alloys

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***Abstract*—A series of Cox-Au1-x granular alloys was grown using pulsed-current electrodeposition on polyimide substrates. The relationship between the giant magnetoresistance effect, saturation magnetization, and grain size was examined as the deposition current density and annealing temperatures were changed. A maximum magnetoresistance (MR) ratio of 4.5 % was obtained. The magnetization and ferromagnetic grain size of the as-deposited and annealed alloy films were examined against the deposition current density. The saturation magnetization decreased as the composition of Au was increased for both the alloys deposited at the current densities of 1 and 5 mA/cm2. The total magnetic moment further decreased on annealing. The grain size is found to be highly influenced by both the deposition current density and temperature. A low temperature magnetization measurement suggested that the ferromagnetic grain size decreases with increase in current densities. For the annealed samples, the ferromagnetic grain sizes were found to be dispersed in the Au matrix with different diameters. The decrease in magnetic moments with annealing is correlated with the grain size of the Co and its distribution in the gold matrix. The MR ratio increased with the increase in the deposition current density as a result of the formation of smaller grain size at higher deposition current densities.**

***Index Terms*—Co-Au granular alloys, giant magnetoresistance effect, saturation magnetization, ferromagnetic grains**

I. Introduction

Magnetic alloys with interesting magnetotransport and magnetic properties have been the subject of intense investigations due to their significance in fundamental physics [1]-[5] and application in electronics [6], [7] and in the automobile [8], [9], and biomedical sectors [10]-[13]. Many novel nanomaterials have been developed from the combination of ferromagnetic 3-d transition metals and non-magnetic group IB metals at an atomic scale [6], [14]-[20], and more are expected to emerge as the research continues. Usually, 3-d transition ferromagnetic metals exhibit MR effect by virtue of their nature as these materials possess interesting electronic configurations where the 4s sub-shells are occupied leaving behind their 3-d shells unfilled. This phenomenon we can understand from the electronic configurations of these metals. On the application of an magnetic field, these 3-d transition metals exhibit interesting anisotropic magnetoresistance effect, which is a change in electrical resistance, where there is a difference between the MR values when the magnetic field is applied parallel and perpendicular to the direction of current [3]. The degree of anisotropy in transition metals is a function of magnetic moment and the angle that it makes with the direction of current. The mechanism of AMR is considered to be due to the interactions of 3-d and 4-s electrons of the ferromagnetic metals [7]. On the other hand, when we combine 3-d transition metals with the group IB non-magnetic metals, they form magnetic multilayers, alloys, and nanoparticles that exhibit GMR effect. What makes the GMR effect different from the AMR effect is its magnitude, which is relatively much larger than the AMR effect reported so far [7], [21]. Also, the magnitude of GMR is independent of the direction of current or applied magnetic field [22]. The mechanism of GMR effect in Co-Au is considered to be due to the interaction of 4s electrons of Au and 3d electrons of Co [3], [23]. For practical alloys, not only should we consider the spin alignments along the cross section of the film but also consider the spin arrangements along the surface of the film [22], [23].

Most of these granular alloys have been grown using non-aqueous methods such as sputtering [24], molecular beam epitaxy [25], e-beam evaporation [26], melt spun [27], and mechanical alloying [14]. However, the relationship between various preparation conditions and giant magnetoresistance (GMR) effect, a large change in electrical resistance at the low applied magnetic field, and magnetic properties has not been clear yet.

Mathematically, the percentage change in electrical resistance due to the applied magnetic field is defined as:

$$MR\% = \frac{[R(H) - R(H_0)]}{R(H_0)} \times 100 \quad (1)$$

where $R(H)$ is the resistance of the film as a function of the magnetic field, H, and $R(H_0)$ is the resistance of the film at zero applied field.

Pulsed-current electrodeposition is one of the preferred electrochemical methods in which magnetic alloys can be grown via reducing the reaction of metal ions from a single electrolyte [28]-[32]. It has many merits over non-aqueous deposition methods. For example, pulse electrodeposition is a room temperature technology, which offers precision in growth on an atomic scale. The pulse electroplating set-up is usually simple. It is also one of the cost effective deposition methods as compared to its high vacuum depositing counterparts. The electrical and magnetic properties can be easily manipulated by controlling the film composition (i.e., pulse amplitude) and thicknesses (i.e., pulse widths) of either Co or Au layers on the atomic scale. Also, it does not require

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extensive training to deposit films using pulsed-current electrochemical deposition method [33].

The magnetic properties of the Co-Au alloys are influenced by the size of the Co grains and their separation from Au grains [7]. The mean Co grain size is estimated as:

$$K_A v = 25 k_B T_B \quad (2)$$

Where K_A is the magnetic anisotropy energy density constant for face-centered cubic Co, v is the volume of a super paramagnetic Co grain corresponding to the blocking temperature T_B, and k_B is the Boltzmann's constant. The blocking temperature was obtained from the zero-field-cooled (ZFC) curves measured by radio frequency-Superconducting Quantum Interface Device (rf-SQUID) meter. This measurement is extremely sensitive to grain interactions and provides a good technique for investigating the energy barrier distribution [34].

Some of the earliest studies on the Co-Au alloys prepared via pulsed-current electrodeposition in aqueous electrolytes have examined their microstructure, magnetization, and magnetoresistance effects [35], [36]. Guo et al. [35] reported room temperature MR of 0.5 % for the as-deposited Co-Au alloys and of up to 1.5 % when annealed at 573 K. They found that the grain size of Co increased with the increase of annealing temperature. Previous studies [37], [38] have shown GMR values of up to 4 % for the as-deposited Co/Au multilayers prepared using pulsed-current electrodeposition in the cyanide solution and demonstrated how closely GMR and magnetic properties are correlated with the layer thickness and composition in the as-deposited Co/Ag and Co/Cu nanostructures [38], [39]. However, it is not yet fully clear to what extent deposition current density, annealing, and layer thickness have an effect on the grain size and on the electrical and magnetic properties of Co-Au alloys.

Therefore, this study describes the preparation parameters of Co-Au granular alloys grown from a cyanide solution on polyimide substrates using constant-current pulsed electrodeposition. A relationship between the MR ratio, saturation magnetization, and composition of Co, and grain size in the alloys has been investigated. We observed that both the deposition current density and annealing have appreciable effect on the GMR, saturation magnetization, and grain size of Co in the Au matrix.

II. Experimental Procedures

Co-Au alloys were prepared using computer-controlled pulsed-current electrodeposition method on 15-nm Cu (111) layers e-beam evaporated on to polyimide substrates. The circuit used for pulsed-current electrodeposition is described in [33]. The electrolyte consisted of cobalt sulphate [$CoSO_4 \cdot 7H_2O$], potassium gold cyanide [$KAu(CN)_2$], sodium citrate [$Na_3C_6H_5O_7 \cdot 2H_2O$], and sodium chloride [NaCl] [38]. The composition of Co in the electrolyte was changed while keeping the composition of Au constant. In this way, the Co:Au ratio could be varied. The total composition of the electrolyte was varied from 10 to 98 at % Co, while keeping the $KAu(CN)_2$, $Na_3C_6H_5O_7 \cdot 2H_2O$, and NaCl constant. The electrochemical parameters are listed in Table 1. The purpose of adding NaCl was to enhance the conductivity of the electrolyte. The $Na_3C_6H_5O_7 \cdot 2H_2O$ was used to improve processing conditions and material properties [40]. Different current densities in the range of 0.1 to 5 mA/cm^2 were tested to deposit pure Au and Co-rich films. Only the results of alloy films deposited at the current densities of 1 and 5 mA/cm^2 are presented here.

TABLE I: Electrochemical Parameters

Constituents	g/l
$CoSO_4$.7H2O	28-39
$KAu(CN)_2$	15-25
$Na_3C_6H_5O_7.2H_2O$	76
NaCl	2
Current density	1-10 mA/cm^2
pH	4-6
Temperature	50 ± 1 ^{0}C

The alloy films were electrodeposited from an unstirred, cyanide bath, the temperature of which was maintained at 50 ± 1 ^{0}C in a temperature-controlled water bath, under dc galvanostatic conditions by employing two electrodes in the single electrolyte. The pulsed-current was supplied and controlled by a programmable microcomputer, digital to analog convertor, and a constant current circuit [33]. A pure Co plate was used as the anode electrode with the sample as the cathode maintained at the distance of 2 cm. After deposition, films were cleaned by double distilled water and dried, and immediately wrapped up in paraffin wax paper to avoid from getting oxidized.

The composition of Co and Au was determined using atomic flame emission spectroscopy and verified using energy dispersive x-ray analysis in a scanning electron microscope. Magnetoresistance measurements were carried out using a standard four-probe DC technique with an excitation current of 3.0 mA. The magnetic field was applied along the parallel and perpendicular to the direction to current. The contacts consisted of nickel (5-15 % of phosphorus) probes. The details of the measurement scheme are given in [41], [42]. All of the measured magnetoresistance values were negative and the absolute values of the transverse MR are presented here. Magnetization measurements were carried out at room temperature using a vibrating sample magnetometer and rf-SQUID meter at low temperature. The films were heat treated at 573 K for 30 minutes. The saturation magnetization of the as-deposited and annealed alloys was estimated from the measured magnetization curves. The temperature dependence of zero-field-cooled (ZFC) and field-cooled (FC) magnetizations for the as-deposited and annealed alloy film was studied using an rf-SQUID meter at the magnetic field of 50 Oe.

III. Results and Discussions

A. Compositional Analysis

Fig. 1 shows a correlation between the Co concentration (Co at %) in the electrodeposited film and Co composition (Co at %) in the electrolyte at the current densities of 1 and 5 mA/cm^2, respectively. The composition relationship between the deposited film and electrolyte was not linear. Hence it can be concluded that the deposition was of regular type [43]. We observed that the less noble element, i.e., Co, is less readily deposited as compared to Au. The Co concentration in the deposited alloy film is lower at the lesser concentration in the electrolyte. However, the concentration increases abruptly for electrolyte compositions in excess of 50 at % Co. At the current density of 1 mA/cm^2, the curve does not saturate even

with the increasing Co composition in the solution. However, at the current density of 5 mA/cm^2, the Co composition in the film approached to the Co composition in the electrolyte. Also, the concentration of Au atoms in solution was very small as compared to Co. Hence the deposition of Au atoms seems to be under the diffusion control [43].

TABLE II: ELECTROCHEMICAL COMPOSITION

Electrolyte	Bath Composition (at % of Co)					
(g/l)	10	30	50	70	90	98
$CoSO_4$	0.35	1.1	2.55	5.85	22	92.5
$KAu(CN)_2$	2.5	2.5	2.5	2.5	2.5	2.5
NaCl	5	5	5	5	5	5
$Na_3C_6H_5O_7$	5	5	5	5	5	5

The composition of Co was changed while keeping the composition of Au, NaCl, and $Na_3C_6H_5O_7$ constant. The amount of $Na_3C_6H_5O_7$ and NaCl was kept constant to 5 g/l. The electrochemical parameters are shown in Table 2. The Co concentration in the electrolyte was changed while keeping the concentration of Au constant: Thus, the ratio between the Co and Au changed. The inset in Fig. 1 demonstrates the correlation between the Co composition in the Co-Au alloys and the deposition current density. It shows that the composition of Co in the film drastically increases with the increase in current density.

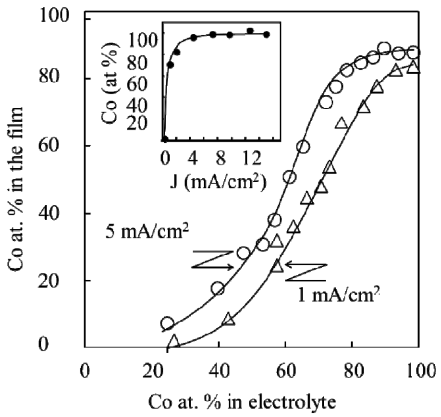

Fig. 1. Co concentration in the deposited alloy versus Co concentration in the electrolyte as the composition of Co in the electrolyte is changed from 10 to 98 at %. Open triangles (Δ) correspond to the current density (J) of 1 mA/cm^2 and open circles (O) correspond to the current density of 5 mA/cm^2. The inset in it shows the extent to which Co composition (at %) in the deposited alloy film increases with the deposition current density as it is increased from 0 to 16 mA/cm^2.

B. Composition Dependence of Magnetoresistance Ratio

Fig. 2 shows the dependence of the MR ratio on the Co concentration for Co-Au single layers deposited at current densities of 1 and 5 mA/cm^2, and measured at room temperature and within an magnetic field of 21 kOe. The maximum MR ratio of the films deposited at 1 mA/cm^2 was 2 %. With an increase in current density to 5 mA/cm^2, the MR ratio increased appreciably for all Co concentrations in the film. The maximum MR ratio was 4.5 % and the concentration of the maximum MR ratio shifted slightly towards the lower Co concentrations. The maximum MR ratio appeared within a narrow concentration range of ferromagnetic atoms even at varying current densities.

The increase in the MR ratio near 30 at % Co seems to be due to the optimum size and distribution of Co grains and inter-granular separation [44], [37]. The low MR values below 30 at% Co can be ascribed to a relatively low concentration of ferromagnetic components leading to fewer magnetic and non-magnetic interfacial scattering sites. The decrease in MR values above 45 at % Co seems to be due to the decrease in spin-dependent scattering, which is mainly responsible for the GMR effect [3], [22], [23]. The increase in the MR ratio with increasing deposition current density is because of a change in microstructure of the film [22], [38]. The current density, composition, and the maximum MR ratio obtained from the experiment are summarized in Table 3.

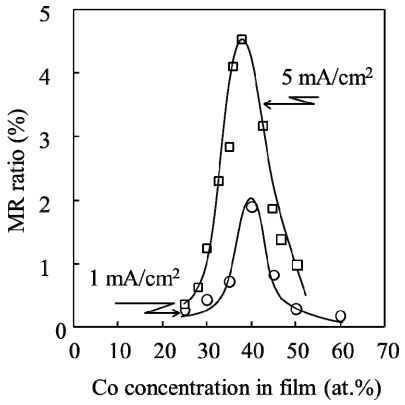

Fig. 2. The MR ratio is plotted as a function of Co concentration (at %) in the deposited alloy film as the current density is changed from 1 to 5 mA/cm^2. Open squares (□) correspond to the MR ratio measured at the current density of 5 mA/cm^2 and open circles (O) correspond to the MR ratio curve measured at the current density of 1 mA/cm^2.

TABLE III: PHYSICAL PARAMETERS

Current Density (J/m^2)	Composition (Co at %)	MR ratio (%)
1	0~60	2
5	0~60	4.5

C. Composition Dependence of Saturation Magnetization

Fig. 3 illustrates the composition dependence of the saturation magnetization for the as-deposited and annealed Co-Au alloy films. We observed that the magnetization decreased as the Co concentration was changed for both types of alloys deposited at the current density of 1 and 5 mA/cm^2. In both cases, the magnetization decreased with the increase of Au at % in the film. The trend of decreasing magnetization with the increasing Au composition is consistent to the results observed for Ni-Cu and Ni-Zn alloys [23]. The decrease in saturation magnetization with the increase of interaction of 4s electrons of Au with the 3d electrons of Co is considered to be the main factor in minimizing the overall magnetic moment of Co in the Co-Au alloys as the composition of Au is increased. This explanation holds well with the explanation given in [23]. Also, there was a sharp decrease in magnetization with the increase in current density. Additional investigations are needed to answer the phenomena behind a sharp decrease of magnetization with the increase of current density. For this purpose, results of rf-SQUID meter will be presented in the later section. It seems that the Co-Au alloys electrodeposited at higher current densities either formed smaller grain sizes or formed solid solution. Similar results have been reported for other ferromagnetic films as well [45], [34]. The saturation magnetization of Co-Au alloys further decreased with the

increase of annealing temperature due to the decrease in Co grain size, increase in Au grain size, and the change in the separation between the Co grains.

The magnetization also deviated from the simple dilution law: It decreased with increasing Au concentration and vanished below 30 at % Co. The saturation magnetization decreases sharply with the increase of Au at % composition. However, the decrease is more abrupt for the alloys deposited at 5 mA/cm^2 as compared to 1 mA/cm^2.

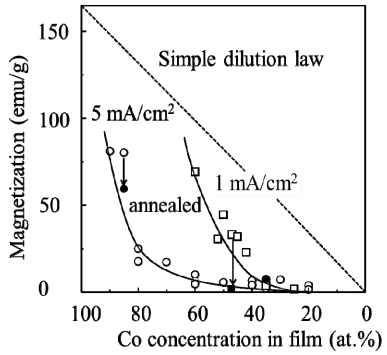

Fig. 3. Saturation magnetization plotted as the concentration of Co is changed from 0 to 100% for the as-deposited and annealed (573 K for 30 minutes) Co-Au alloys. Open squares (□) and open circles (O) represent magnetization measured at the current densities of 1 and 5 mA/cm^2, respectively. Similarly, closed rectangles (■) and closed circles (●) represent saturation magnetization of the annealed Co-Au alloys. The dotted straight line drawn from the magnetization of 160 emu/g to zero represents simple dilution law.

D. Microstructural Analysis

X-ray diffraction spectra of $Co_{35}Au_{65}$ alloy films deposited at the current density of 1 mA/cm^2 and 5 mA/cm^2 obtained from Rigaku $CuK\alpha$ X-ray diffractometer are illustrated in Fig. 4, where the prominent broad peak at $2\theta = 44.25^0$ corresponds to (111) fcc-Co crystallites and is less intense compared to the peak due to (111) Au: The pure Au (111) and Au (200) peaks are located at $2\theta = 38.2^0$ and $2\theta = 44.37^0$, respectively. The peak height of Au (111) reduces significantly when the deposition current density increased to 5 mA/ cm^2 from the applied current density of 1 mA/cm^2. The peak at $2\theta = 43.25^0$ corresponds to the Cu substrate.

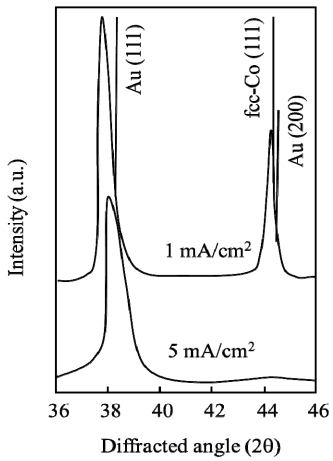

Fig. 4. X-ray diffraction patterns for the as-deposited $Co_{35}Au_{65}$ alloys as a function of the current density (1 and 5 mA/cm^2) as the diffraction angle, 2θ is changed from 36 to 46^0.\

The presence of strong peaks of Au indicates that Au here served as the film matrix whereas Co grains are dispersed as small clusters throughout the film: The grain size of the Co was determined using a low temperature rf-SQUID magnetization measurement, and is described below. This result is consistent with the recently reported transmission electron microscopy image of Co-Au nano-composites that shows a clear formation of Co grains with the diameter in the range of 1- 3.3 nm dispersed in the Au matrix [35]. This is also somewhat analogous to the recently reported X-ray diffraction analysis, which shows the formation of Au grains in Co_{68} Au_{32} granular alloys [11].

E. Estimation of Cobalt Grain Size

Fig. 5 shows the temperature dependence of zero-field-cooled (ZFC) and field-cooled (FC) magnetization for the Co-Au alloy films in the (a) as-deposited and (b) annealed states. During the magnetization measurement, the samples were first cooled to 5 K at the magnetic field of 50 Oe. However, the magnetic moment was measured during the heating cycle in the range of 5 K to 300 K. In the as-deposited sample, a broad peak was observed near the temperature of 65 K at the deposition current density of 5 mA/cm^2.

If the peak of the ZFC curve is the mean blocking temperature observed for super-paramagnetic behavior, the existence of the peak at the lower temperature suggests that the very fine grains of the ferromagnetic Co or Co-Au alloy phase may have been precipitated and they had a wide distribution in size [35]. Since the Co-rich magnetic grains were present in the matrix as a fcc phase, taking K_A equal to 5.5×10^{17} eV/cm^3 (1 eV = 1.6×10^{-19} C) corresponding to the bulk fcc-Co, the volume of the grain could be calculated. Assuming spherical shapes of the grains, the estimated mean cobalt grain diameter corresponding to the blocking temperature was estimated to be approximately 4 nm. We also calculated the grain size of Co for the samples deposited at the current density of 1 mA/cm^2, and found that the grain are larger when deposited at the lower current densities.

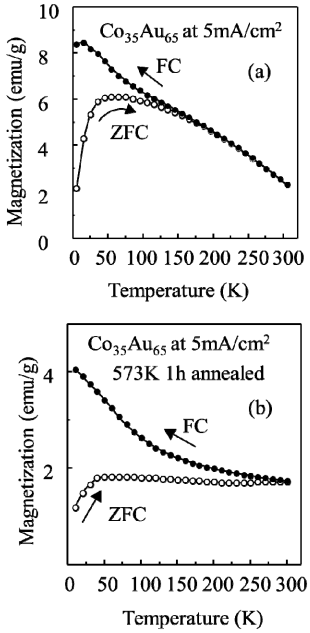

Fig. 5. The saturation magnetization of (a) as deposited and (b) annealed $Co_{35}Au_{65}$ alloys as the temperature is changed from 5 to 298 K: Closed circles (●) indicate the field cooled (FC) curve when the temperature is changed from 298 K to 5 K at 50 Oe and open circles (o) indicate the zero-field cooled (ZFC) when the temperature is increased 5 K to 298 K at zero applied magnetic field.

As shown in Fig. 5(b), unlike the broad peak observed at 65 K for as-deposited alloys (Fig. 5(a)), the peak for the annealed Co-Au alloy is not significant. The peak extends all the way to the room temperature, i.e. the bifurcation of ZFC and FC curves occurs at only close to the room temperature (~ 300 K) for the annealed film. This suggests that the grain size of Co had a range of blocking temperatures. This also suggests a change in the size and interaction effects of Co grains in the Au matrix with a wide distribution range. These results are consistent with the results of the low temperature magnetizations of Co-Cu [30] and Co-Ag [34] films**.** It is possible that the small cobalt grains present in Co-Au films deposited at the current density of 5 mA/cm^2 contributed to the larger MR values for these films [44].

IV. Conclusions

Co-Au granular alloys have been prepared by using computer-controlled pulsed-current electrodeposition. The MR ratio and grain size in the film largely depended on the current density. The maximum MR ratio of $Co_{35}Au_{65}$ alloy deposited at the current density of 5 mA/cm^2 was 4.5 % at 40 at % Co. The temperature dependence of the magnetization curves at low temperature suggested the formation of fine grains exhibiting super-paramagnetic behavior. It is considered that the presence of small Co grains in the as-deposited films at 5 mA/cm^2 contributed to the larger MR values for these films. Also, the temperature dependence of magnetization of annealed $Co_{35}Au_{65}$ alloys at 573 K suggests that a decrease in magnetization correlates with the size and distribution of Co grains and their separation from Au grains in the Co-Au matrix. This work is significant because of its usefulness in developing electronic devices based in magnetic technology for the application in the automobile and biomedical sectors.

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