

## ELECTRODEPOSITION OF Co/Cu MAGNETIC MULTILAYERS AND THEIR CHARACTERIZATION USING AUGER DEPTH AND LINE PROFILES AND MAGNETIZATION MEASUREMENTS

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### ABSTRACT

An automated system was built for the electrodeposition of binary multilayers using two electrochemical cells, which allows the number and thickness of successive layers to be programmed and executed by moving the substrate and immersing it into the corresponding bath, with predetermined current and deposition time values. This system was used for the fabrication of planar magnetic Co/Cu (magnetic/non-magnetic) multilayers with layers of nanometric thickness. These multilayers were characterized by obtaining Auger depth composition profiles, as well as line composition profiles in images of the footprint left on the sample surface by sputtering with a focused Ar<sup>+</sup> beam. For the magnetization measurements in the flat samples, as a function of the in-plane applied magnetic field, an 860 nm thick Co film deposited on a Cu substrate was prepared as a reference sample. Three other samples were prepared with this same amount of Co, but distributed into 20 layers of 43 nm each, separated respectively from each other by 22, 11 and 5 nm-thick Cu layers. The obtained magnetization results suggests with an antiferromagnetic coupling between successive Co layers when the separation between them is 5 nm of Cu.

**Keywords:** Electrodeposition, Multilayers, Nanowires, Auger-profiles

### ELECTRODEPOSICIÓN DE MULTICAPAS MAGNÉTICAS Co/Cu Y SU CARACTERIZACIÓN MEDIANTE MEDIDAS DE MAGNETIZACIÓN, MICROSCOPIA ELECTRÓNICA Y PERFILES AUGER

### RESUMEN

Se construyó un sistema automatizado para la electrodeposición de multicapas utilizando dos celdas electroquímicas, el cual permite programar y ejecutar el número y espesor de las sucesivas capas desplazando el sustrato para sumergirlo en el baño correspondiente, con valores predeterminados de corriente y de tiempo. Este sistema se utilizó para la elaboración de multicapas magnéticas planas de Co/Cu (magnética/no-magnética) con capas de espesor nanométrico. Estas multicapas fueron caracterizadas mediante la obtención de perfiles Auger de composición en profundidad, así como de perfiles lineales en las huellas de decapado dejadas tras el bombardeo con un haz de Ar<sup>+</sup>. Para las medidas de magnetización en las muestras planas, como función del campo magnético aplicado en el plano, se elaboró como muestra de referencia una película de Co de 860 nm de espesor depositada sobre un sustrato de Cu. Se elaboraron otras tres muestras con esta misma cantidad de Co, pero distribuida en 20 capas de 43 nm cada una, separadas entre sí respectivamente por capas de Cu de 22, 11 y 5 nm. Los resultados de magnetización obtenidos sugieren un acoplamiento antiferromagnético entre sucesivas capas de Co cuando la separación entre ellas es de 5 nm de Cu.

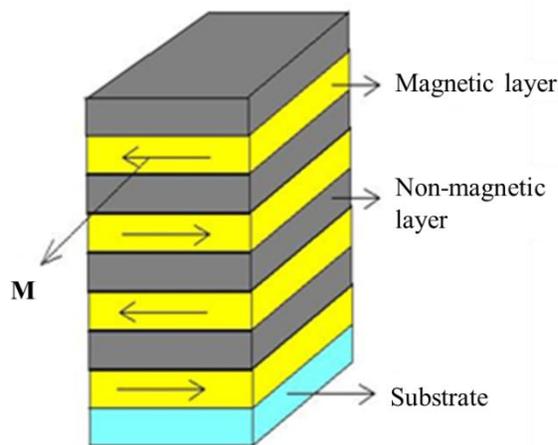
**Palabras clave:** Electrodeposición, Multicapas, Nanoalambres, Perfiles-Auger

### INTRODUCTION

The Giant Magneto-Resistance (GMR) phenomenon [1] occurs in multilayered systems where nanometric-thick layers of ferromagnetic materials with magnetization **M** alternate with also nanometric-thick layers of non-

magnetic materials in such a way that, in the absence of an applied external magnetic field, neighboring magnetic layers are antiferromagnetically coupled as shown schematically in Figure 1. The methods commonly used for the fabrication of these systems are magnetron

sputtering and molecular beam epitaxy, methods adequate for the fabrication of flat multilayers but not appropriate for samples with other shapes. It is of interest to find more versatile and less



**Figure 1.** Schematic drawing of an antiferromagnetically coupled multilayer system.

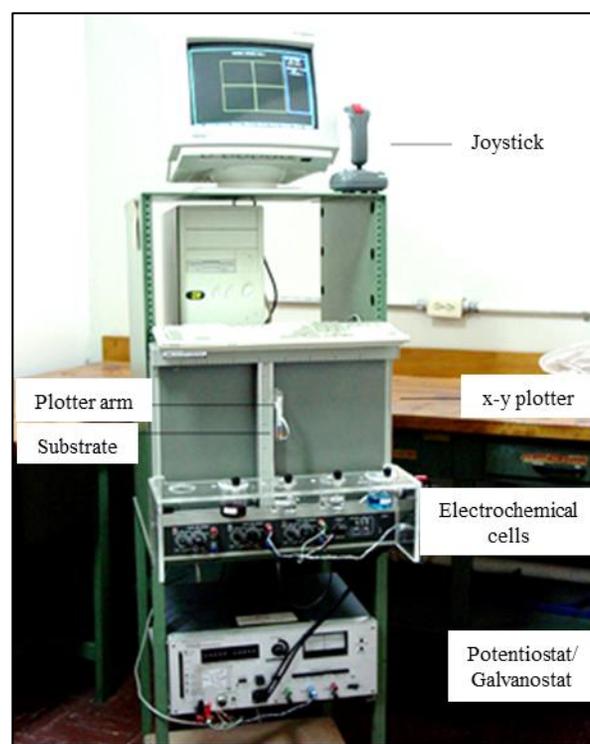
expensive techniques for the production of magnetic nanosystems. One of such techniques is electrochemical deposition, a method known for a long time to produce protective coatings at industrial level and that has been adapted for the fabrication of multilayers showing the GMR phenomenon [2]. In this work we have used electrodeposition to produce nanostructured Co/Cu multilayers as planar samples, where the Co layers are the magnetic layers, separated by the non-magnetic Cu layers. For such a purpose an automated system was built for the electrodeposition of binary planar multilayers that were then characterized by Auger Electron Spectroscopy and magnetization measurements.

## MATERIALS AND METHODS

### *Automated Electrodeposition System*

For electrodepositing the Co/Cu multilayers the double bath method was used, with one electrochemical cell of  $\text{Co}^{2+}$  ions and platinum counter-electrode and the other with  $\text{Cu}^{2+}$  ions and copper counter-electrode. An automated system controlled by an old PC was built that

allows programming the number and thickness of the different layers and alternating the use of each of the cells by moving the substrate and immersing it in the corresponding bath, with predetermined current and deposition time values [3]. Figure 2 shows a photograph of this Automated Electrodeposition System. It consists of three main elements: i) the potentiostat/galvanostat (PAR 362), ii) the sample manipulator (x-y plotter modified to replace the pen by 1  $\text{cm}^2$  working electrode) and iii) the control computer with 12-bit A/D and D/A cards and a joystick for programming the sample motion in order to immerse it in each cell and to wash it in distilled water after each layer deposition.



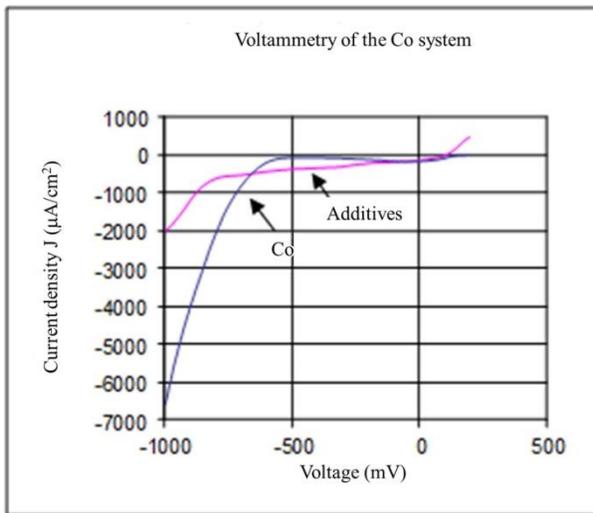
**Figure 2.** Automated Electrodeposition System.

Once the sequence of motions required to deposit a bilayer is recorded with the joystick, a computer program allows the fabrication of the desired multilayer, specifying ionic species of each bath, the number of repeat cycles  $N$  and the desired current value for each cell, to be used in the constant current mode for

electrodeposition. In this mode the thickness of each metallic layer is determined by the time during which the preset current for each cell is maintained. We assume that the measured current is mainly due to the reduction of the species of interest, which allows us to use Faraday's Law to determine the nominal thickness of each layer. The substrates used for the planar multilayers were 1 cm<sup>2</sup> flat Cu plates made from commercial sheets used for the manufacture of printed circuits.

*Electrochemical systems*

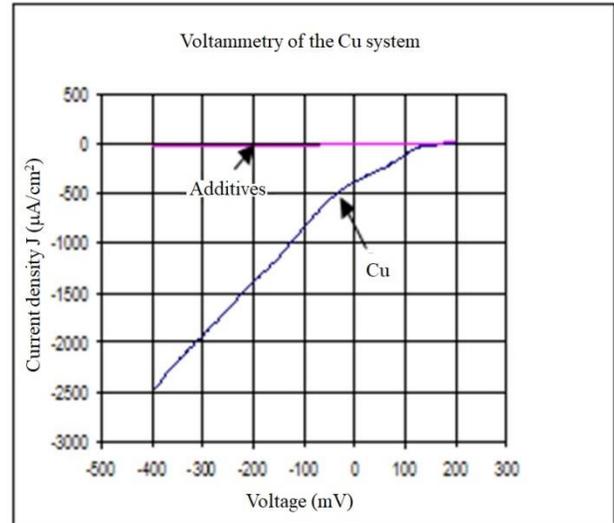
Two electrochemical systems were prepared and characterized by Voltammetry: a) for Co deposition and b) for Cu deposition. For Co deposition the electrochemical systems consists of a 100 ml aqueous solution containing [0.60 M] hydrated cobalt (II) sulfate (CoSO<sub>4</sub>xH<sub>2</sub>O), plus the additives boric acid [0.30 M] (H<sub>3</sub>BO<sub>3</sub>) and [0.65 M] hydrated tri-sodium citrate (Na<sub>3</sub>C<sub>6</sub>H<sub>5</sub>O<sub>7</sub>x2H<sub>2</sub>O), adjusting the pH to 5 with [1M] sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). Figure 3 shows the Voltammetry characterization of this system.



**Figure 3.** Voltammetry graph of the Co bath.

For Cu deposition the electrochemical systems consists of a 100 ml aqueous solution containing [0.15 M] hydrated copper (II) sulfate (CuSO<sub>4</sub>xH<sub>2</sub>O) plus the

additives [0.30 M] boric acid (H<sub>3</sub>BO<sub>3</sub>) and [0.65 M] tri-sodium citrate hydrate (Na<sub>3</sub>C<sub>6</sub>H<sub>5</sub>O<sub>7</sub>x2H<sub>2</sub>O), adjusting the pH to 5 with [1M] sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). Figure 4 shows the Voltammetry characterization of this system.



**Figure 4.** Voltammetry graph of the Cu bath.

From the Voltammetry graphs are chosen the values of the electric current to be used for the electrodeposition.

*Auger Electron Spectroscopy characterization*

The flat multilayers systems were characterized by Auger depth and line profiles using a Scanning Auger Microprobe with a Hemispherical Deflection Analyzer. Depth profiles were obtained by sputtering with 3 keV Ar<sup>+</sup>. Auger spectra and secondary electron images were obtained using a 5 keV electron beam.

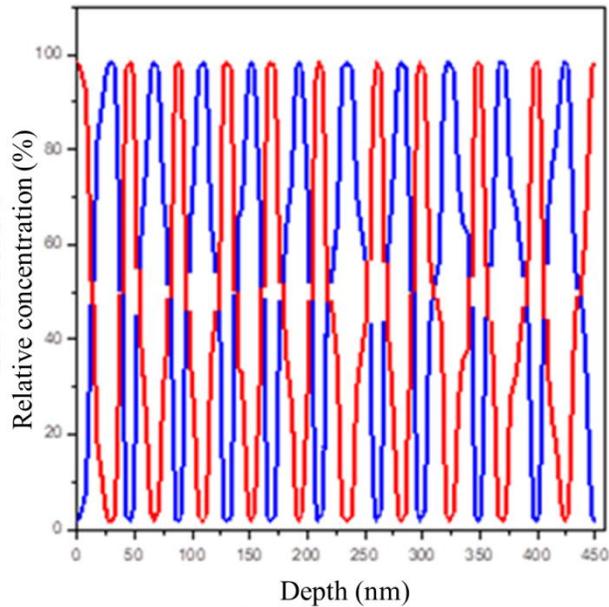
*Magnetization measurements*

Magnetization graphs M(H) were obtained using a Faraday Balance with an applied magnetic field H parallel to the sample surface, with varying intensities from 0.05 to 0.74 T.

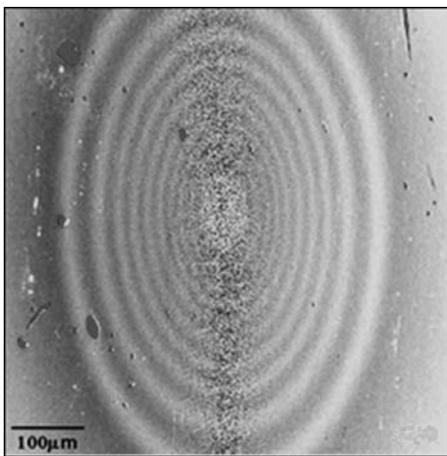
**RESULTS AND DISCUSSION**

As an example of the results obtained, we present in Figure 5 an Auger depth composition profile of the

10[Cu(29nm)/Co(79nm)]/(Cu) multilayer, obtained by bombarding the sample with 3 keV Ar<sup>+</sup> at 14 nm/min sputtering speed. In Figure 6 we show the corresponding sputtering footprint left after bombardment, a secondary electron image of the affected area, which allows us to expose and visualize the different layers of the system [4]. The footprint image appears elliptical because the sample was tilted 55° in order to face the Argon gun.

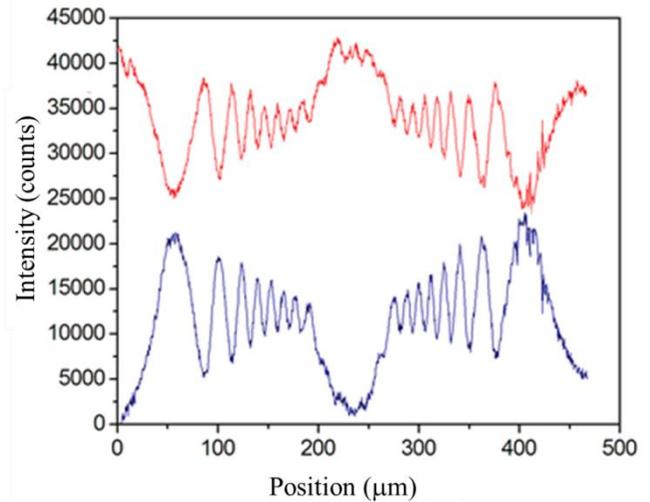


**Figure 5.** Auger depth composition profile of the 10[Cu(29nm)/Co(79nm)]/(Cu) planar multilayer. Red line: Cu. Blue line: Co.



**Figure 6.** Secondary electron image of the sputtering footprint of the 10[Cu(29nm)/Co(79nm)]/(Cu) planar multilayer. Dark bands: Co. Bright bands: Cu.

The relative intensity of the rings depends on the local atomic number. The light rings correspond to the heavy element (Cu) and the dark ones to the light element (Co). This image can be used to identify the different layers by obtaining a line composition Auger profile like the one shown in Figure 7.

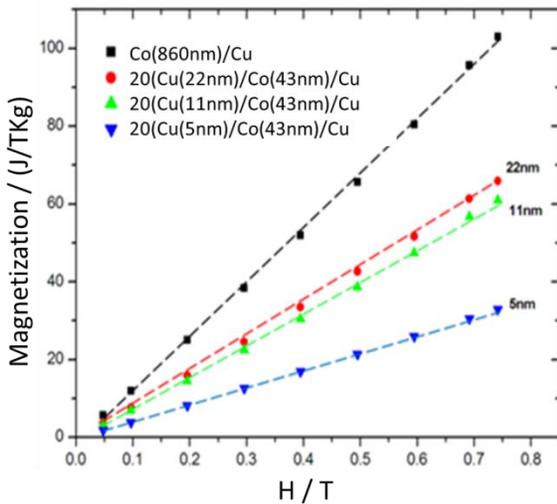


**Figure 7.** Auger line composition profile through the center of Figure 6. Red line: Cu. Blue line: Co.

Once the production method of the Co/Cu multilayers was controlled, we wanted to observe their magnetic behavior as a function of the thickness of the separating Cu non-magnetic layers, which can affect the magnetic coupling between adjacent ferromagnetic Co layers. As a reference sample, a 860 nm thick Co layer was deposited on a Cu substrate and its magnetization  $M(H)$  was measured as a function of the applied in-plane magnetic field  $H$ . This same amount of Co was distributed into 20 layers of 43 nm each, separated from each other by Cu layers of variable thicknesses. In this way three other samples were prepared, with Cu separator layers of 22, 11 and 5 nm respectively. The corresponding  $M(H)$  curves are presented in Figure 8. We see that in the four cases the magnetic response to the applied field is linear, not reaching saturation with our maximum available field of 0.74T. With 22 and 11 nm Cu separator thickness the magnetization values are similar and smaller than the value corresponding to the reference single layer Co film,

but with the thinner 5 nm Cu separator layer the magnetization values are noticeable smaller, what could

indicate an antiferromagnetic coupling between the Co layers.



**Figure 8.** Comparison of the M(H) curves between a reference single layer Co film and multilayer Co/Cu films with Cu layers of different thickness.

## CONCLUSIONS

An automatic electrodeposition system for the efficient fabrication of planar bimetallic multilayer systems was designed. This electrodeposition system was successfully used for the fabrication of different Co/Cu multilayer samples.

The Scanning Auger Microprobe proves to be a very convenient instrument for the characterization of flat multilayer samples, by the generation of Auger depth profiles, secondary electron images of sputtering footprints and linear Auger profiles along positions in the sample corresponding to these images.

The Co layers in the N[Cu(5 nm)/Co(43 nm)]/Cu seems to be coupled antiferromagnetically.

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