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1995 J. Phys.: Condens. Matter 7 2081

(http://iopscience.iop.org/0953-8984/7/10/014)

J. Phys.: Condens. Matter 7 (1995) 2081–2085. Printed in the UK

$Co_{65}Fe_{35}$/Ag alloy and multilayer growth checked by *in situ* resistance

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Received 10 October 1994, in final form 29 November 1994

Abstract. We present an *in situ* resistance study of evaporated $Co_{65}Fe_{35}$/Ag alloys, which present a giant magnetoresistance (GMR) effect. We have observed the influence of the substrate and evaluated the initial perturbed layer in coevaporated alloys, in multilayers with thick layers, and in multilayers with very thin layers. When very thin 'layers' of each metal are deposited, a granular structure is observed. This particular preparation mode allows us to observe, at the beginning of the deposition, a preferential sticking of the $Co_{65}Fe_{35}$ alloy on the substrate rather than on the already deposited Ag. This result shows the dependence of the granular structure on the sample thickness and corroborates GMR results.

1. Introduction

Numerous studies are devoted to alloys and multilayers made of immiscible magnetic and non-magnetic metals because of their magnetoresistance properties. In particular, granular $Ag_{1-x}(Co_{65}Fe_{35})_x$ alloys prepared by coevaporation have been studied in our laboratory [1, 2]. A magnetoresistance coefficient of 87% at $T = 4.2$ K under a field of 60 kOe has been observed in an $Ag_{75}(Co_{65}Fe_{35})_{25}$ alloy prepared at 100 °C. *In situ* resistance measurements were used to check the sticking of the metal on different substrates [2–7]; they were also used to observe structural changes occurring during growth or annealing [3–5]. In the present paper, we focus on measurements of *in situ* resistance of Ag/$Co_{65}Fe_{35}$ coevaporated alloys and multilayers to observe the substrate effect and the respective role of the metals. A sample prepared by successive deposition of very thin 'layers' of $Co_{65}Fe_{35}$ and Ag (1.1 Å and 5 Å respectively) was also studied. This sample presents a GMR effect close to that of the alloy. In this case, *in situ* resistance measurements may give an insight into the alloy deposition mode.

2. Sample preparation

The substrate, prepared for resistivity measurements, consists of a thin film of adhesive kapton pasted on Corning glass (size, 20×5 mm^2), which can be removed from the glass for magnetization measurements. Electrodes of Cr and Au (size, 5×5 mm^2, Cr thickness, 500 Å; Au thickness, 1500 Å) are deposited by evaporation at 100 °C on each end of the substrate. The size of the measured sample is then 10×5 mm^2. The resistance measurement threads are fixed on the electrodes with a conductor silicone paste charged with Ag. This paste is then polymerized by annealing for 1 h at 190 °C.

0953-8984/95/102081+05$19.50

The deposited thickness is measured with quartz oscillation sensors and monitored by a controller (Leybold Infincon IC/4 plus) providing a thickness resolution of 0.01 Å for a unit material density and a measurement frequency of 10 Hz. The electrical resistance is measured by a conventional four-point method with a DC current. A computerized system records the electrical resistance and the deposited thickness every second.

A 100 Å thick layer of amorphous silicon is deposited on the kapton film at 100 °C, just before the deposition of the samples. The pressure during the deposition is 10^{-8} Torr. The samples are prepared by evaporation of Ag (99.99%) and $Co_{65}Fe_{35}$ (99.9%) using an electron beam gun for the $Co_{65}Fe_{35}$ alloy and a Joule heating crucible for Ag. The composition of the samples presented here is $Ag_{75}(Co_{65}Fe_{35})_{25}$ (at.%). The substrate temperature is maintained at 100 °C during the growth of each film. The deposition rate is 1.8 Å s^{-1} for the codeposited alloy and 0.7 Å s^{-1} and 0.2 Å s^{-1} for the multilayer samples with a $Co_{65}Fe_{35}$ layer of 10 Å and 1.1 Å respectively.

Using x-ray $\theta/2\theta$ scans, we observe a strong preferential orientation of the Ag grains with (111) planes parallel to the substrate. The grain growth occurs then in a direction perpendicular to the densest planes of the structure.

3. Experimental results

The conductance σ is presented in figure 1 as a function of the deposited thickness t for (1) a codeposited alloy, (2) multilayers with thin layers, and (3) multilayers with thick layers. After an initial stage where σ is zero, two regimes are observed: a transitory regime where σ increases steeply and then a permanent regime.

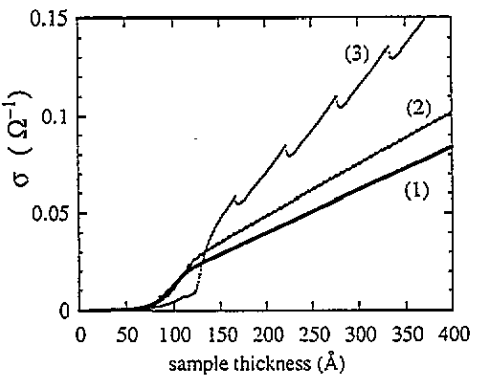

Figure 1. The general evolution of the *in situ* conductance σ as a function of sample thickness for $(Co_{65}Fe_{35})_{25}Ag_{75}$ samples deposited at 100 °C on amorphous silicon. (1) codeposited alloy; (2) multilayer sample with thin layers; $t_{Ag} = 5$ Å and $t_{Co_{65}Fe_{35}} = 1.1$ Å; (3) multilayer sample with thick layers, $t_{Ag} = 45.5$ Å and $t_{Co_{65}Fe_{35}} = 10$ Å.

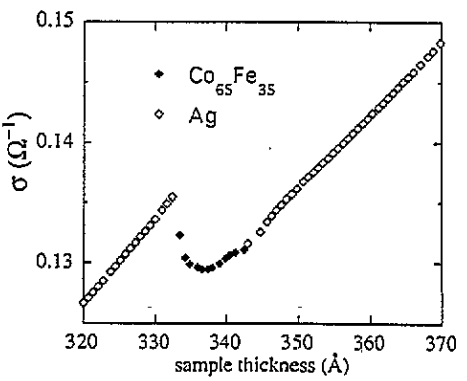

Figure 2. *In situ* conductance σ in the permanent regime as a function of the deposited thickness for a $(Co_{65}Fe_{35})_{25}Ag_{75}$ multilayer sample with $t_{Ag} = 45.5$ Å and $t_{Co_{65}Fe_{35}} = 10$ Å.

For codeposited alloy, σ is zero for a thickness smaller than 70 Å, then increases steeply up to 120 Å, and then varies linearly with the thickness of the films. This variation of σ can be interpreted as follows. At the deposition temperature of 100 °C, no interdiffusion of

Ag or $Co_{65}Fe_{35}$ in the silicon substrate can be expected. Thus the variation of σ with t can be attributed to the mobility of Ag, Co, and Fe atoms on the silicon surface. In the first stage, for $t < 70$ Å, isolated islands are formed on the surface and σ remains equal to zero. Percolation is observed for $t \approx 70$ Å so the conductance steeply increases as the available space between the islands is filled. In the third stage of the variation of the conductance, σ grows linearly with the thickness t. As shown in a previous paper, percolation occurs for a larger thickness on glass or kapton substrates [2].

In the case of multilayers, the same influence of the substrate is qualitatively recovered with a larger perturbed layer for thick multilayers (≈ 170 Å).

In the permanent regime, the non-monotonic variation of σ with t, which appears clearly for the thick-layer sample (figure 1(3)), corresponds to the alternation of the metals as shown in figure 2. A decrease of the conductance is observed when the $Co_{65}Fe_{35}$ alloy is deposited on Ag, a minimum occurs for a $Co_{65}Fe_{35}$ thickness of 5 Å, and then σ increases again. We recall that Ag and $Co_{65}Fe_{35}$ are practically immiscible, so interdiffusion is very limited. The decrease of the conductance at the beginning of the $Co_{65}Fe_{35}$ deposition is then due to an increase of the diffuse scattering of the Ag conduction electrons at the sample surface. When the available surface of Ag is filled, the conductance reaches a minimum and increases again with the increasing contribution of $Co_{65}Fe_{35}$ conduction electrons. The occurrence of the minimum at 5 Å, corresponding roughly to two monolayers, may indicate that the growth of $Co_{65}Fe_{35}$ on the Ag surface is not strictly a layer by layer growth but involves two or three atomic layers simultaneously. In order to establish a precise correlation between *in situ* resistance measurements and growth modes, it would be necessary to study the growth on a monocrystalline surface and to couple this study with *in situ* characterization measurements [6]. Our results are reminiscent of the observations made with Co deposited on Cu [7] or Cr deposited on Fe [8]. In contrast, when covering $Co_{65}Fe_{35}$ with Ag, we observe an increase of the conductance from the outset of the Ag deposition.

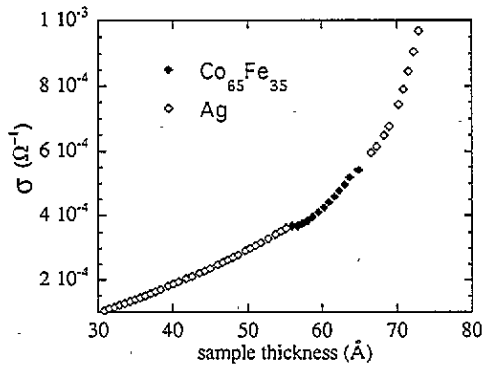

Figure 3. *In situ* conductance σ, at the beginning of the deposition, as a function of the deposited thickness for a $(Co_{65}Fe_{35})_{25}Ag_{75}$ multilayer sample with $t_{Ag} = 45.5$ Å and $t_{Co_{65}Fe_{35}} = 10$ Å.

During the deposition of the first layers, unlike the case in the permanent regime, we do not observe any decrease of the conductance when $Co_{65}Fe_{35}$ is deposited, as shown in figure 3. This particular behaviour of $Co_{65}Fe_{35}$ during the sticking of the metals on the substrate is detailed below for the sample with thin layers.

The GMR response of multilayers with very thin layers (of the order of the atomic scale) is close to that of the alloy (75% with respect to 87% at 4.2 K under a field of

60 kOe). Moreover, magnetization measurements show that the low-field magnetization of the sample with thin layers, after a zero-field cooling, presents a peak as granular alloys do (to be published). Using the high-field approximation of the Langevin function [1], we determine a particle radius of 18 ± 2 Å, which is much larger than the $Co_{65}Fe_{35}$ layer thickness. Thus it can be concluded that this sample presents a granular structure as do the codeposited alloys.

In situ conductance measurements clearly show different effects of the $Co_{65}Fe_{35}$ in the different stages of the sample sticking on the substrate (figure 1(2)), described above for the alloy.

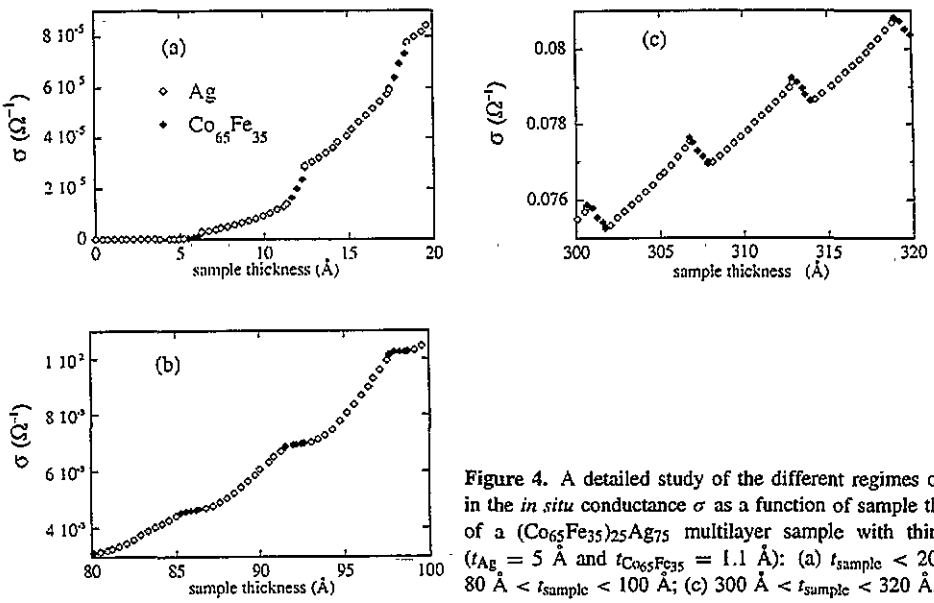

Figure 4. A detailed study of the different regimes observed in the *in situ* conductance σ as a function of sample thickness of a $(Co_{65}Fe_{35})_{25}Ag_{75}$ multilayer sample with thin layers ($t_{Ag} = 5$ Å and $t_{Co_{65}Fe_{35}} = 1.1$ Å): (a) $t_{sample} < 20$ Å; (b) 80 Å $< t_{sample} < 100$ Å; (c) 300 Å $< t_{sample} < 320$ Å.

The evolution of the conductance of this type of multilayer is plotted in figure 4(a) at the beginning of the deposition. During the completion of the first Ag layer (5 Å), $\sigma = 0$, which corresponds to the formation of isolated islands. Then, an immediate increase of the conductance is observed when $Co_{65}Fe_{35}$ is deposited. For the subsequent layers, we can note a stronger increase of the conductance when $Co_{65}Fe_{35}$ is deposited than when Ag is deposited. This indicates that the $Co_{65}Fe_{35}$ alloy improves the connection between the Ag grains. It is expected to preferentially settle on the substrate between the Ag islands as illustrated in figure 5(a).

Just after the metallic percolation, for a sample thickness larger than 80 Å, we observe a saturation of the conductance when the $Co_{65}Fe_{35}$ alloy is deposited, as shown in figure 4(b).

In the permanent regime (figure 4(c)) we observe a decrease of conductance when covering with $Co_{65}Fe_{35}$. This shows that $Co_{65}Fe_{35}$ no longer induces the coalescence of Ag grains, but settles on the Ag surface and increases the surface diffuse scattering of Ag conduction electrons as was previously observed with thicker multilayers. This is illustrated in figure 5(b).

The granular structure of the sample is induced by the diffusion of the Co and Fe atoms on the Ag surface. We can therefore assume that the successive deposition of very thin 'layers' is a way of simulating the deposition of the alloy. In this case, the tendency of $Co_{65}Fe_{35}$ alloy to settle between the Ag grains may explain the sample thickness effect

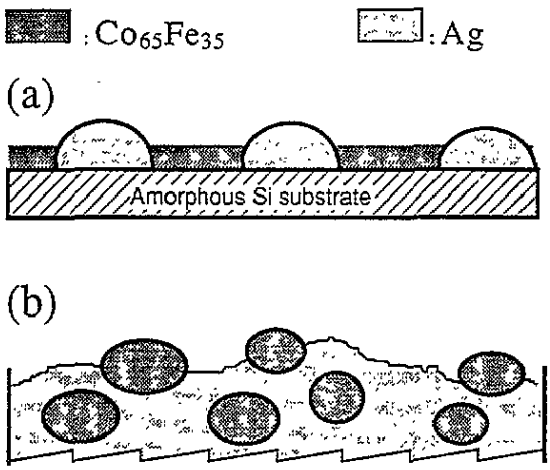

Figure 5. An illustration of the $Co_{65}Fe_{35}$ and Ag deposition mode of $(Co_{65}Fe_{35})_{25}Ag_{75}$ multilayer samples with $t_{Ag} = 5$ Å and $t_{Co_{65}Fe_{35}} = 1.1$ Å (a) at the beginning of the deposition and (b) after metallic percolation.

observed on the GMR, which has been related to a decreasing size of particle with decreasing thickness [2, 9].

4. Conclusion

The growth of $Co_{65}Fe_{35}$/Ag alloys and multilayers has been followed using *in situ* resistance measurements. The homogeneous growth of the alloy occurs after a deposited thickness of roughly 120 Å. When very thin layers of each material are deposited, the granular structure of the codeposited alloy is recovered. This allows us to show that the $Co_{65}Fe_{35}$ alloy settles between the Ag grains at the beginning of the deposition whereas it forms grains at the Ag surface when the homogeneous growth is reached. This shows the sample thickness dependence of the granular structure.

Acknowledgment

We are grateful to Professor M Gerl for his critical reading of the manuscript.

References

[1] Bellouard C, George B and Marchal G 1994 *J. Phys.: Condens. Matter* **6** 7239
[2] Bellouard C, George B and Marchal G 1995 *Proc. Int. Conf. on Magnetic Films and Surfaces (Dusseldorf, 1994); J. Magn. Magn. Mater.* at press
[3] Pariset C 1976 *Thesis* University of Paris Sud
[4] Dufour C, Cherifi K, Bruson A, Marchal G and Mangin Ph 1991 *Phys. Status Solidi* a **125** 561
[5] Marliere C 1986 *Thin Solid Films* **136** 181
[6] Jalochowski M and Bauer E 1988 *Phys. Rev.* B **37** 8622
[7] Eckl Th, Reiss G, Brückl H and Hoffmann H 1994 *J. Appl. Phys.* **75** 362
[8] Jacob M, Reiss G, Brückl H and Hoffmann H 1992 *Phys. Rev.* B **46** 11 208
[9] Mitchell J R and Berkowitz A E 1994 *J. Appl. Phys.* **75** 6616