CHARACTERISATION OF CO–FE ALLOYS ELECTRODEPOSITED WITH MAGNETIC FIELD SUPERIMPOSITION

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Introduction. During the last decade, a decisive advance has been made in the field of elaboration and development of soft magnetic films [1, 2, 3] for industrial use, *e.g.* in the heads of reading – writing. We study the influence of the magnetic field on the composition, structure, morphology and magnetic properties of Co-Fe films worked out by electroplating. The effect of the magnetic field is confirmed by X-ray diffraction (XRD) and by scanning electronic miscroscopy (SEM).

1. Experimental. Co-Fe alloys were electrodeposited in a classical thermostated (25°C) three-electrode cell. The electrolyte contained cobalt sulfate (0.2 M), iron(II) sulfate (0.025 M), boric acid (0.4 M), sodium sulfate (0.5 M) and sodium chloride (0.3 M). The pH was adjusted to 3 by sulfuric acid addition. The deposit was obtained on a ∅10 mm (copper or titanium) working electrode, a saturated sulfate mercury electrode (SSE) was used as reference electrode and a large area platinum as counter electrode. Potentiostatic depositions were conducted with a Tacussel potentiostat-galvanostat PGZ 301 during 30 min. When a magnetic field was superimposed, the cell was put into the gap of an electromagnet (Drusch EAM 20G) controlled by a Hall probe to deliver a constant homogeneous horizontal magnetic field. The working electrode was placed horizontally upwards in the parallel magnetic field configuration or vertically in the perpendicular one. XRD experiments were performed with a BRUKER D8 diffractometer equipped with a copper anticathode ($\lambda_{\mathrm{CuK}\alpha}$ = 54056 Å), SEM micrographies and quantitative analyses of the deposits were obtained by means of a JEOL JSM 6460LA miscroscope coupled with a EDS JEL 1300 microprobe.

Alloy composition analyses have been carried out by optic emission spectroscopy by means of an inductive coupled plasma (ICP-AES) and have been confirmed by EDS measurements.

2. Parallel magnetic field. Current-potential curves for Co-Fe alloys are reported on Fig. 1. For each curve, a predeposit was firstly done by potentiostatic method at – 1.4 V/ESS for 10 minutes without magnetic field, then the curve was recorded with a low scan speed (2 mV/s). It can be seen that the magnetic field has an effect on the curves by slightly increasing the amplitude of the cathodic current. This effect arises from the MHD effect on the involved electroactive species: Fe^{2+}, Co^{2+} and H^{+}. Because one (or more) species, depending on the applied potential, can be involved in the convective diffusion process, it is not obvious to predict the current variation that could be either increased or decreased [4] by the MHD convection.

For −1.4 V/SSE applied potential, all the electrodeposited alloys have the same composition, whatever the applied magnetic field: the iron atomic amount is ≈ 14%. It is the opposite in Ni-Fe alloy electrodeposition [4] wherein nickel species are not mass-transport controlled and wherein the iron contain increases with the magnetic field intensity.

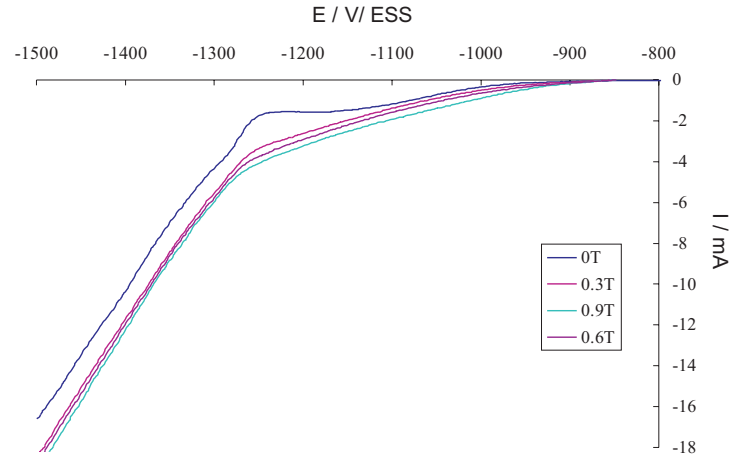

Fig. 1. Current-potential curves for Co-Fe system. $CoSO_4$ 0.2 M. $FeSO_4$ 0.025 M. Na_2SO_4 0.5 M. NaCl 0.3 M. H_3BO_3 0.4 M. pH = 3. $T = 25°C$. The magnetic field is parallel to the horizontal upwards electrode ($\varnothing = 10$ mm). Scan speed = 2 mV/s.

Notwithstanding this atomic constant level, the XR diagrams highlight drastic modifications for alloys according to the magnitude of the superimposed parallel magnetic field (Fig. 2).

Without forced convection, the diagram exhibits a *fcc* Co spectrum (index JCPDS no. 150803) with a [111] texture. Only a small signal (for $2\theta = 45,2°$) attests the presence of another phase. This line can be attributed to the [110] reflection of the *fcc* Co_7Fe_3 phase.

When a magnetic field is superimposed, the diagram exhibits more and more the Co_7Fe_3 phase lines as those of Co gradually disappear. For the highest magnetic fields used during this work, the very intense signal for $2\theta = 45.2°$ is a piece of evidence that the Co_7Fe_3 phase exhibits a specific [110] texture.

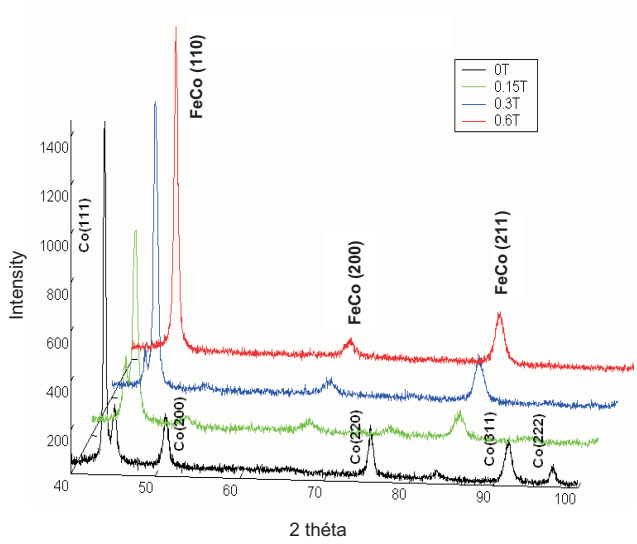

Fig. 2. XRD diagrams for Co-Fe alloys electrodeposited at −1.4 V/SSE. The magnetic field was parallel to the horizontal electode. Same bath as Fig. 1.

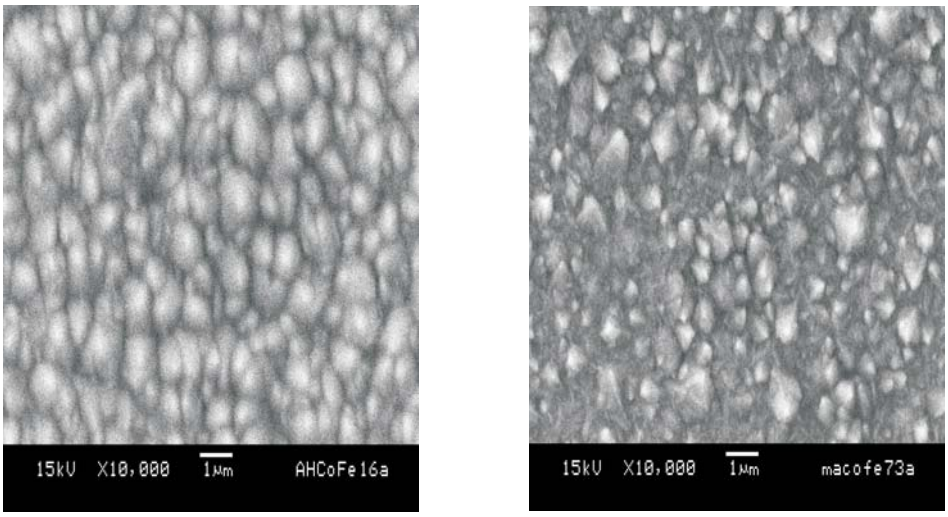

Fig. 3. SEM micrographies for Co–Fe alloys electrodeposited at −1.4 V/SSE. The magnetic field was parallel to the horizontal electrode. Same bath as Fig. 1.

SEM micrographies for two deposits obtained at the same applied potential and with or without magnetic field (Fig. 3) exhibit homogeneous surfaces with spots that have no special geometrical form, but the spot size dramatically decreases when magnetic field is applied. This result is typical for the levelling effect of magnetic field [5].

3. Perpendicular magnetic field. For the same electrolytic conditions and a low magnetic field perpendicular to the electrode surface, it is impossible to obtain any thick deposit for magnetic fields above 0.3 T because dendritic growth arises without any preferential direction. If we compare the XR diagrams for electrodeposited alloys under 0.3 T magnetic field superimposition (Fig. 4), we can notice that the effect of the perpendicular magnetic field is lower than the

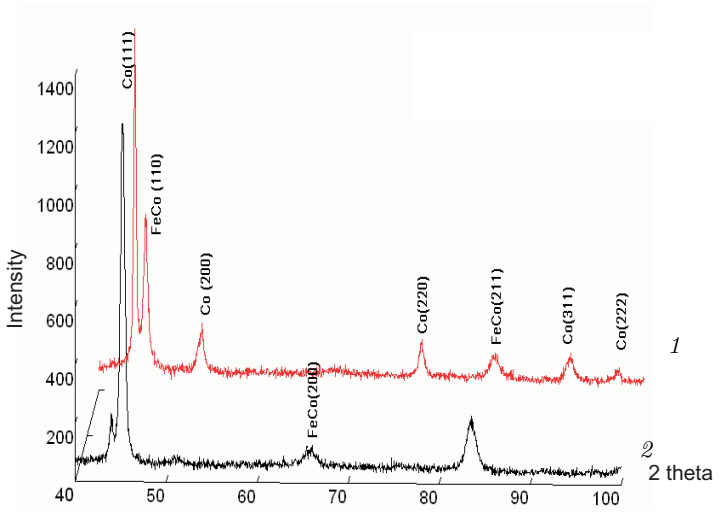

Fig. 4. XRD diagrams for Co–Fe alloys deposited at 1.4 V/SSE. **B** = 0.3 T. *1*: **B** perpendicular, *2*: **B** parallel.

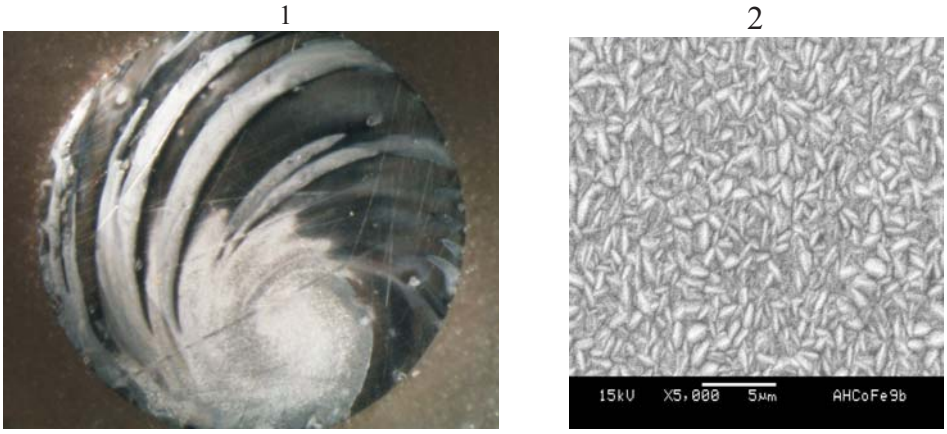

Fig. 5. Aspect of the CoFe alloy electrodeposited under a 0.3 T perpendicular magnetic field. 1) optic photography. 2) SEM micrography of a white zone of the deposit.

parallel one. In the first case, the *fcc* Co structure is always existing and only one line for the Co_7Fe_3 structure can be identified.

The most important effect can be seen on Fig. 5 where vortex effects can be seen when a 0.3 T field is applied. If the spot size and form are quite different in this case and are responsible for the coloured aspect of the deposit, quantitative analyses by EDS have led to the same ratio between iron and cobalt wherever the point the analyses have been realized. Such an effect has to be deeply investigated because hydrogen evolution, micro MHD or ferro and paramagnetic effects can be suspected.

4. Conclusion. The electrodeposition of CoFe alloys under magnetic field superimposition has been investigated with magnetic fields either parallel or perpendicular to the surface. Magnetic fields less than 1 T do not change the atomic composition of the alloys but, depending on the magnetic direction, drastic changes on the morphology and the texture of the deposits have been highlighted. If some effects can be explained by classical MHD, some further investigations have still to be undertaken.

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