

# Magnetic composites CoNi–barium ferrite prepared by electrodeposition

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## Abstract

CoNi–barium ferrite magnetic composites have been prepared by using a chloride electroplating bath. The use of a cationic surfactant and a vigorous stirring of solution by means of a rod stirrer have been the key to assure a significant insertion of particles in the deposits. This incorporation was confirmed by chemical analysis, cross-section images and X-ray diffraction results. A maximum value of 12 wt.% of barium ferrite was obtained. Anomalous codeposition of CoNi was maintained, although the cobalt percentage was enhanced by the presence of the cationic surfactant. A clear enlargement of composite coercivity was obtained as a consequence of micrometric hard magnetic particles insertion in the deposits.

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## 1. Introduction

Recently, a significant effort has been employed in the research and development on micro-electromechanical systems (MEMS) actuators in academic and industrial fields. Different actuation principles have been reported. The electromagnetic one is an attractive alternative to other actuations because offers some advantages as its reversible effect, high speed and long-distance transmission of an intense force [1,2]. In addition, low fields are enough, so there is low power consumption. Most of the magnetic micro-actuators developed so far are made from soft-magnetic materials [3–7]. However, hard-magnetic materials are also desirable in some applications (micro-sized motors, actuators, mini-pumps, bidirectional push-pull micro-actuators and micro-actuators driven by off-chips coils) [8,9]. The most common hard-magnetic materials are CoPt, FePt and rare earths as Nd<sub>2</sub>Fe<sub>14</sub>B or SmCo<sub>5</sub>

[10–12]. However, the preparation of MEMS devices containing these materials has been mainly limited by technical difficulties in producing accurate permanent magnets at microscale [13]. The manufacture of permanent magnets to be used in micro-devices presents an important drawback because the process is time-consuming and expensive.

The challenge lies on developing a deposition process of hard-magnetic materials compatible with MEMS processing. Several micro-machining techniques such as screen-printing, sputtering and electrochemical deposition have been applied to deposit and integrate permanent magnets in order to MEMS devices. However, screen printing is usually limited to millimetre scale and the magnetic properties of thick sputtered magnets are worse than those of the bulk material. Electrodeposition is the most versatile technique due to its low cost and its capability of forming micro-nanometric features. Unfortunately, readily available and reliable deposition processes lack because the magnetic properties of the deposits do not satisfy the required characteristics. Moreover, the codeposition of rare earth magnetic thin films implies the use of organic solvents and

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the application of high negative potentials. This causes the damage of some photoresist electroplating molds [14].

Thus, it is important to explore new deposition techniques that retain the advantages offered by electrochemical deposition while providing hard magnets with better magnetic properties for magnetic actuation. In order to achieve this objective the electrochemical preparation of magnetic composites embedded in a metallic matrix is proposed.

The aim of the present work is to explore the electrodeposition technique capability of incorporating barium ferrite in cobalt–nickel deposits obtained from an optimized plating bath [15,16]. The barium ferrite embedding is expected to increase the coercivity of the magnetic films. The influence of deposition potential, hydrodynamic conditions or surfactant presence on morphology, composition and structure of the composites will be analysed. Moreover, the variation of both coercivity and magnetization saturation of the CoNi films induced by the presence of magnetic particles, will be studied.

## 2. Experimental

Magnetic composites preparation has been performed in a conventional three-electrode cell of 0.25 dm<sup>3</sup>, at a constant temperature of 55 °C. Solutions contained CoCl<sub>2</sub>, NiCl<sub>2</sub>, boric acid and saccharine, all of analytical grade. A cationic surfactant (dodecyltrimethylammonium chloride – DTAC) has been used in some experiments. One micron-sized barium ferrites from Alfa Aesar have been used as magnetic particles. Solutions were freshly prepared with water which was first doubly distilled and then treated with a Millipore Milli Q system. The solution pH was maintained at 3.0. Solutions were de-aerated by argon bubbling before each experiment and maintained under argon atmosphere during it.

Electrodeposition has been carried out using a micro-computer-controlled potentiostat/galvanostat Autolab with PGSTAT30 equipment and GPES software. Electrochemical study and deposits preparation was performed using a silicon/ Ti(1000 Å)/Ni(500 Å) electrode supplied by IMB-CNM.CSIC (Centro Nacional de Microelectrónica). The silicon-based substrates were cleaned with acetone followed by ethanol and rinsed in water before deposition. The reference electrode was an Ag/AgCl/1 mol dm<sup>-3</sup> NaCl electrode. All potentials are referred to this electrode. The counter electrode was a platinum spiral.

Voltammetric experiments were carried out under quiescent or stirring conditions at 50 mV s<sup>-1</sup>, scanning initially from –500 mV towards negative potentials. A single cycle was run in cyclic voltammetric experiments. Deposits were prepared under different stirring regimes using a rod stirrer placed in the centre of the cell. The relative position of the electrodes was maintained in all conditions.

Hitachi S 2300 and Leica Stereoscan S-360 scanning electron microscopes have been used to analyse surface morphology and deposit section. Also Hitachi S-4100 Field

Emission scanning electron microscope (FE-SEM) was used in some cases. Deposits composition was determined by inductively coupled plasma optical emission spectrometry (ICP-OES) with a Perkin Elmer Optima 3200 RL. Different solutions were used to dissolve the CoNi coatings: 2% HNO<sub>3</sub> and aqua regia. CoNi–barium ferrite composites were dissolved in aqua regia.

Structure of deposits were studied by means of X-ray diffraction (XRD), using a Siemens D-500 equipment. Diffractograms were obtained in the 10–100° 2θ range with a step range of 0.03° and a measuring time of 8 s per step.

Magnetic measurements were taken in a SQUID magnetometer at room temperature.

Roughness (rms) of the coatings was measured using a white-light interferometer from Zygo Corporation.

## 3. Results

The possibility of embedding magnetic particles in CoNi deposits was analysed as a function of the barium ferrite concentration, hydrodynamic conditions and the presence of a surfactant in the bath. The electroplating bath consisted of a 0.2 mol dm<sup>-3</sup> CoCl<sub>2</sub> + 0.9 mol dm<sup>-3</sup> NiCl<sub>2</sub> + 30 g dm<sup>-3</sup> boric acid + 0.7 g dm<sup>-3</sup> saccharine, pH = 3 solution, at 55 °C (basic bath). Different concentrations of cationic surfactant (DTAC) were tested.

CoNi deposits of 4–8 μm thick were prepared as reference, always maintaining the analogous deposition conditions than in presence of barium ferrite particles. The electrolytic bath was usually mechanically stirred at high rotation speeds. For each experimental condition, a previous voltammetric study was performed to select the deposition potential adequate to assure a similar deposition rate.

Fig. 1 shows the voltammetric curves corresponding to cobalt–nickel deposition process in absence or presence

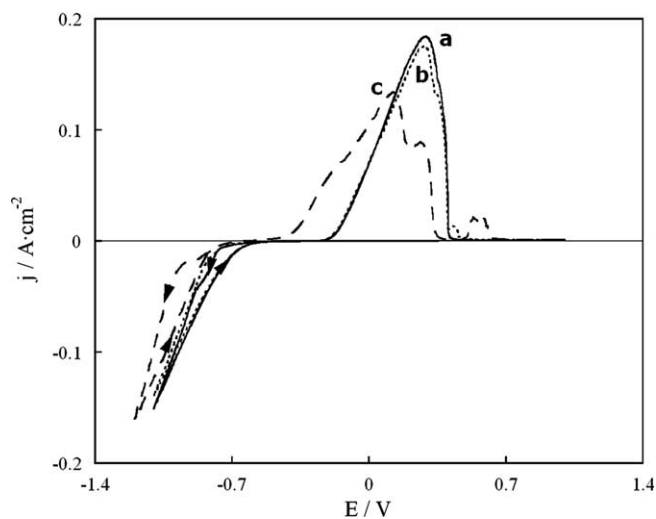


Fig. 1. Cyclic voltammograms at 50 mV s<sup>-1</sup> of 0.9 mol dm<sup>-3</sup> NiCl<sub>2</sub> + 0.2 mol dm<sup>-3</sup> CoCl<sub>2</sub> + 30 g dm<sup>-3</sup> H<sub>3</sub>BO<sub>3</sub> + 0.7 g dm<sup>-3</sup> saccharine (basic bath) + *x* g dm<sup>-3</sup> DTAC + *y* g dm<sup>-3</sup> BaFe<sub>12</sub>O<sub>19</sub>: (a) *x* = 0, *y* = 0; (b) *x* = 0, *y* = 100; (c) *x* = 0.46, *y* = 100. Stirring at ω = 3000 rpm.

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