

Influence of a magnetic field during the CoNi electrodeposition in the presence of magnetic nanoparticles

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Abstract

CoNi electrodeposition in the presence of barium ferrite nanoparticles has been performed in a bath containing a cationic surfactant, in order to prepare magnetic composites. Magnetic particles did not significantly influence the metallic deposition process. The application of a magnetic field during the electrodeposition over silicon-base substrates has been also analysed. The application of a magnet attached to the silicon/seed-layer substrate inhibited the onset of the deposition process as a consequence of the hindrance of the cathode by the magnetic particles surrounding the electrode. However, once the deposition process begins, the applied magnetic field clearly favours the nanoparticles incorporation by modulating the deposition potential or current density. Barium ferrite percentage of the deposits increased at low current densities or deposition potentials.

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1. Introduction

In the last years, the preparation of composites has been attained increasing interest due to the improved properties of nanostructured materials. The properties of these nanostructured materials (corrosion resistance, hardness, mechanical resistance, magnetic behaviour, magnetoresistance or electrocatalytic properties) will depend on the amount of particles incorporated into the matrix, their size and distribution.

One of the recent interests in composites field is the preparation of magnetic composites. Some of the performed studies aim to prepare magnetic composites by embedding magnetic particles in a non-metallic matrix [1–6]. Other authors deal with the incorporation of magnetic particles into a metallic non-ferromagnetic matrix [7–11] in order to modulate their magnetic properties. There are

also studies focused in the preparation of magnetic composites with ferromagnetic matrix [12–16]. In this case, the purpose is usually the enhancement of the hard-magnetic behaviour of the material for its implementation in microelectromechanical systems (MEMS) devices.

Different techniques have been used for magnetic composites preparation: milling methods [1,5,6,12,15], sol–gel [2,3], ion implantation [4], dc sputtering [8], RF-sputtering [9,10] or chemical synthesis [11,14]. Electroless [16] has been also tested in the preparation of hard-magnetic composites.

Electroplating has been revealed as a tool of producing composites consisting of inert particles and a metallic matrix for its advantages with regard to other techniques [17,18]. In our laboratory, the incorporation of magnetic particles into a metallic matrix has been performed by means of electrodeposition technique, both for non-ferromagnetic [19] and ferromagnetic matrix [20,21]. The incorporation of barium ferrite microparticles into an alloy matrix has been studied for the

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preparation of permanent magnets compatible with micro electro mechanical systems (MEMS) technology.

Several studies have been performed in order to study the influence of electrodeposition parameters on particles incorporation in a metallic matrix, but these studies refer to incorporation of non-magnetic particles. Searson et al. [22,23] describe a kinetic model for electrochemical deposition of Ni–Al₂O₃ nanocomposites. Robertson et al. [24] analyse the influence of several parameters in the Al₂O₃ incorporation in Ni matrix and Walsh et al. [17] review the recent literature on the electrodeposition of metallic coatings containing nanosized non-magnetic particles.

However, the influence of a magnetic field during composites electrodeposition when magnetic particles are used is still not well understood. The aim of the present work is to analyse the electrodeposition process of CoNi system in the presence of hard-magnetic barium ferrite nanoparticles when a magnetic field is applied and also to compare the process in the absence of the field. The selected electrodeposition bath is similar to the one used for alloy-magnetic microparticles composite preparation [21] and it is developed from a basic CoNi bath which leads to fine-grained deposits, with uniform composition at appreciable deposition rate, useful to prepare CoNi microstructures over silicon-based substrates [25,26]. The percentage of magnetic nanoparticles incorporation as a function of the deposition parameters and magnetic field presence will be also analysed.

2. Experimental

CoNi electrodeposition has been performed from a CoNi solution containing 0.9 mol dm⁻³ NiCl₂ + 0.2 mol dm⁻³ CoCl₂ + 30 g dm⁻³ H₃BO₃ + 0.7 g dm⁻³ saccharine. Different concentrations of AZTMAI (4-ethylazobenzene-4'-(oxyethyl)trimethylammonium iodide) (1–5) g dm⁻³ and barium ferrite nanoparticles (2–50) g dm⁻³ were added to the basic solution. The AZTMAI was synthesised in our laboratory as previously described [27]. This surfactant was used to prevent particles agglomeration and to favour the particle-cathode electrostatic attraction. Barium ferrite nanopowder of 99.5%, from Sigma–Aldrich, has been used. Temperature was maintained at 25 °C and pH of solutions was adjusted at 3. All solutions were freshly prepared with water which was first doubly distilled and then treated with a Millipore Milli Q system. Solutions were de-aerated by argon bubbling. Solutions containing nanoparticles were maintained at stirred conditions in the presence of the cationic surfactant during 1 h previous to electrochemical experiments.

Electrochemical experiments were carried out using a microcomputer-controlled potentiostat/galvanostat Autolab with PGSTAT30 equipment and GPES software. A cylindrical three-electrode cell was used. The reference electrode was a Ag/AgCl/1 mol dm⁻³ NaCl electrode. All potentials are referred to this electrode. The counter electrode was a platinum spiral.

Voltammetric curves were recorded under quiescent or stirring conditions at 50 mV s⁻¹. A single cycle was run in cyclic voltammetric experiments.

The study of the deposition process in the presence of the nanoparticles has been performed on both vitreous carbon (Metrohm, 0.0314 cm²) and Si/Ti(100 nm)/Ni(50 nm) electrodes (IMB-CNM, 0.3 cm² of depositing area). Vitreous carbon was polished by using alumina of different grades (3.75 and 1.85 μm) and cleaned ultrasonically for 2 min in water before each experiment. Silicon-based substrates were cleaned with acetone followed by ethanol and rinsed in water before deposition.

Deposits were prepared over the Si/Ti/Ni substrates from different stirring regimes (using a mechanical stirrer) and electrolytic bath composition. A magnet of 350 G was attached to the working electrode in order to analyse the influence of the magnetic field during the electrodeposition.

Morphology of deposits was observed by using Hitachi S 2300 scanning electron microscope. Deposits composition was determined by inductively coupled plasma optical emission spectrometry (ICP-OES) with a Perkin–Elmer Optima 3200 RL, after dissolving the deposits in aqua regia.

Structure of deposits was studied by means of X-ray diffraction (XRD), by using 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.

3. Results and discussion

3.1. Characterisation of barium ferrite nanoparticles

Magnetic and structural characterisation of the selected barium ferrite nanoparticles has been performed. Particles were characterised by means of SQUID, by recording magnetization–magnetic field curves at room temperature. Fig. 1A corroborated the hard-magnetic behaviour of the selected particles, with a saturation magnetization of 40 emu g⁻¹ and coercivity value of 3700 Oe. Some differences in the saturation magnetization were observed respect to the previously used barium ferrite microparticles [20].

The X-ray diffractograms recorded for the magnetic particles show (Fig. 1B) main peaks corresponding to barium hexaferrite structure. Small peaks at 24.1, 33.2, 35.6 and 49.4° 2θ can be assigned to Fe₂O₃ hematite structure. Diffraction peaks corresponding to barium ferrite were wide. The estimation of the crystallite size domain was obtained from X-ray line broadening by using Scherrer's equation. From the full width at half maximum (FWHM) of the reflection peaks, a particle-size ranged between 20 and 35 nm was obtained. Then, X-ray analysis corroborates the nanometric size of the barium ferrite particles.

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