

FABRICATION OF Ni-Co AND Ni-Co/BaFe NANOWIRES IN AAO TEMPLATE

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Abstract. The aim of this paper was the fabrication of Ni-Co and Ni-Co/barium ferrite (BaFe) nanowires in sulfamate electrolyte by using anodic aluminum oxide (AAO) as template. Self-ordered porous AAO membranes have been fabricated and used as templates in order to produce Ni-Co/BaFe nanowires. Morphological and structural studies of nanowires have been performed by Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD), respectively. Energy Dispersive X-Ray Analysis (EDAX) was used for determination of the chemical composition. The nucleation process of nanowires was monitored by galvanostatic analysis. Our investigations showed the formation of the Ni-Co and Ni-Co/BaFe nanowires by incorporating nanoparticles during electrodeposition process. The magnetic investigations have been performed by using Superconducting Quantum Interference Device (SQUID) and the influence of the BaFe particles embedded in Ni-Co wires was revealed.

Key words: porous materials, templates, nanowires, microstructure, magnetic properties.

1. INTRODUCTION

In the last decade, nanosized magnetic materials have been the subject of many studies due to their special physical properties, especially due to their technical applications such as magnetic memories [1]. Some of these applications require hard magnetic materials characterized by coercivity and high remanent magnetization [2]. Several studies have been shown that the magnetic particles, with a proper size similar to those from the magnetic domains, present a significant increase of coercivity as compared to the bulk material [3]. In this particular context an interesting role plays the nanocomposite materials consisting of hard magnetic nanoparticles incorporated within a matrix. This opens a new way to obtain new materials that combine the hard magnetic properties with high saturation magnetization realizable in soft materials [4]. A way to use these

properties of the nanostructures in commercial applications is to include magnetic particles in the ordered magnetic nanowires by electrodeposition technique. In this way, the novel magnetic properties are determined by the shape anisotropy and nanometric dimensions. The template used for this process are essential because induce the model and the characteristics of the nanowires. It is possible to change some specific nanowires feature (diameter, distribution of the nanopores and template thickness) by varying some parameters during the fabrication process of anodic aluminum oxide (AAO) template [5].

Advanced properties and various applications of nanocomposite materials could provide new performance of built products [6]. The improvement in magnetic properties of one nanostructured material is due to the nanoparticles incorporation. The properties of device made of incorporated magnetic nanoparticles increase the information storage capacity [7]. Co and Co-Cu nanowires compose the magnetic devices and solar cells [8]. Nanowires are the most used nanomaterials in nano-sized sensor appliance design. As utility the nanowires could be used in medicine, biology as biosensors, and renewable energy supplies [9]. Also, the inorganic nanowires with excellent optical and electrical properties are used as nanosensors detecting proteins, DNA, molecules etc. In 1988, Baibich has described the magnetoresistive giant (GMR) effect [10]. First attempts to obtain Co-Cu multilayer nanowires in polycarbonate membrane with pore diameter of 40 nm and 10 μm as length are assigned to Piraux [11]. Experimentally, 15% GMR are obtained at room temperature. A comparative study between the nanowires obtained from the following ferromagnetic metals (Co, Ni, Fe electroplated into the polycarbonate porous membrane with 30–50 nm in pore diameter) was achieved. Different magnetic behavior depending on the crystalline anisotropy of Co nanowires was obtained [12, 13].

Nowadays, the electrochemical deposition of the powder from the solution [14] is considered an important process in the surface finishing conferring new functionalities to nanocrystalline materials. The electroplating of alloys from iron-group metals (Fe, Co and Ni) have a resumed interest in fabrication of nanostructured metallic materials because of their better permanent magnetic properties compared to bulk materials [15]. These relatively new materials are amorphous alloys with excellent isotropic properties [16]. The alloy structure presents no long-range order being a general feature for multi-element material [17]. Structural and thermodynamical instability require the selection of an optimal preparation method and working conditions. A wide range of potential applications (*e.g.* high density magnetic memory devices, miniature sensors or field emission display etc.) have also an important role [18–25]. Orinakova *et al.* have studied the theoretical and practical aspects on the electrodeposition of Ni onto non-uniform surface [26]. The microelectromechanical system represents an advantageous example of the combining of mechanical elements, sensors, and electronics on a regular silicon substrate through microfabrication technology [27]. Over the years,

the practical and scientific aspects of these materials and their alloys were concerned the scientists. These nanostructured magnetic materials are studied from the point of view of correlation between the structure and properties having promising practical character [28].

The fabrication of large arrays of ordered, ultra-fine (with dimension of ~ 10 nm or smaller) and monodispersed nanoelements is still a challenge in the material science. Among various methods to obtain nanowires, the direct electrodeposition into the nanopores of some membranes (“template” synthesis) is a very simple, efficient and low cost method [19, 20, 24]. This method is a deposition process into the natural and artificial holes existing in an insulating layer laid a conductor substrate. By using this method can be obtained ordered arrays and nanowires for various applications. In order to improve the nanowires properties there are many factors as: material composition, crystalline state (single crystal, poly-crystal) or amorphous state, single layer or multilayer structure, geometric array etc., as well as properties of nanoporous template.

In order to obtain nanowires by electrodeposition process can be use various nanoporous templates as polycarbonate membranes, nanoporous mica and AAO [29]. Nowadays AAO is used as most common template to fabricate the inorganic template and nanowires. The AAO template is widely used due to its properties, such as: the workability, the compatibility with different materials, ease of manufacturing process and low-cost and highly ordered porous structure [30–32].

One-dimensional (1D) magnetic nanowire arrays have attracted considerable attention in recent years because of their potential applications in high-density magnetic recording media [33–35]. Because of its unique characteristics [36], such as controllable nanopore diameter and length, high pore density, ideal cylindrical pore structure, good mechanical strength, and high thermal stability, the AAO template has become an ideal template to prepare one-dimensional nanowires by many methods. Some of materials used to fill the AAO are: Co [22], Ni, Cu [23], Fe [37], and their alloys [24]. The electrodeposition of Co alloy nanowires with Ni, Fe or Cu is intensely studied due to the special properties of these alloys [38–40]. Notably, the magnetoresistance and giant magnetoresistance (GMR) of these alloys have attracted a great interest [41–42]. It is known that Co is a hard material with variable magnetic properties with the structural parameters such as texture, shape and magneto-crystalline anisotropies [43–44]. Highly ordered Co nanowire arrays with uniform diameter are essential for studying their properties and application in the high density magnetic recording devices in future [45].

The behavior of the AAO template has been studied by many researchers. It has been found that the breakdown of the oxide film and plastic deformation of the aluminum substrate can be suppressed by formation of a porous oxide layer on the surface [46–49]. Also, high current densities have a significant influence on the self-ordering of nanopores for a given anodization potential [50–51].

The aim of this paper was the fabrication of Ni-Co and Ni-Co/BaFe nanowires in sulfamate electrolyte by using AAO as template structures and magnetic characterization of these nanostructures. By using two-step anodization method the Ni-Co nanowires were electrodeposited on nanoporous AAO membrane with 30 nm average diameter. Hexagonal arrangement of nanopores and a separation of 100 nm among wires were observed [19]. The results obtained by means of the magnetometer indicate that the alloy and composite nanowires have a strong magnetic anisotropy.

2. EXPERIMENTAL

2.1. PREPARATION OF THE AAO TEMPLATE

Porous AAO membranes with an ordered hexagonal structure have been obtained by anodic oxidation that occurred in two steps. Initially, the high purity aluminum sheet (99.99%) was degreased in acetone, for 10 minutes and cleaned with distilled water, and then, it was subjected to a mechanical and electrochemical polishing. The last process was conducted in a mixture consisting of perchloric acid, glycerine and ethanol (20:10:70) during 20 minutes.

The anodic oxidation process occurred in cell having a titanium membrane as cathode, an aluminum sheet as anode and a reference electrode of platinum. The first anodic oxidation process was carried out in a 0.3 M oxalic acid solution that was vigorous shake and maintained at 1°C by a HAAKE K10 cryostat. By applying a voltage of 40 V for 4 hours, has been obtained an oxide layer which was removed by immersion in a mixture of 0.2 M CrCrO₃ chromic acid and 0.4 M H₃PO₄ at 65°C for an hour. The second anodic oxidation process was carried out in the same solution at a voltage of 40 V for 8 h and formed on aluminum sheet a porous membrane having nanopores with a diameter of approximately 30 nm. In order to remove any particles formed during the anodic oxidation process, porous membrane was immersed in acetone for 1 h.

At the end of the anodization processes, the aluminum sheet was removed using a 0.1 M CuCl₂ and 5 M HCl solution, and what remained was cleaned with distilled water. In order to open the nanopores of membrane, the oxide barrier layer that remains at aluminum/aluminum oxide interface was removed by dissolving in 1 M H₃PO₄ solution at 40°C for 30 min. During this immersion the upper side of the nanopores was been protected with an enamel.

For electrodeposition of wires, the lower part of the porous membrane was covered with a thin gold layer having a thickness about of 40 nm by Physical Vapor Deposition (PVD). This layer plays a role of the electrical contact of the AAO.

In Fig. 1 a schematic representation of AAO membrane on aluminum sheet filled with nanoparticles, also including the barrier layer is shown.

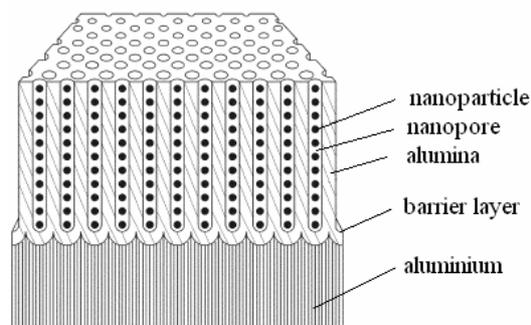


Fig. 1 – Schematic representation of alumina membrane on aluminum sheet filled with nanoparticles including the barrier layer.

2.2. PREPARATION OF NANOWIRES

The electrodeposition of nanowires in AAO template was performed by using a device EG&G Princeton Applied Research, Model 273A. The electrolytic bath for the deposition of Ni-Co nanowires was a mixture of two electrolytes, Ni sulfamate and Co sulfamate with the following compositions: Ni (SO_3NH_2) 1.87 M, H_3BO_3 0.58 M, $\text{NaC}_{12}\text{H}_{25}\text{SO}_4$ $5 \cdot 10^{-4}$ M and Co (SO_3NH_2)₂ 0.09 M respectively. The pH of the electrolytic bath was maintained at a constant value of 4. For preparation of Ni-Co/BaFe nanowires in the above mixture, magnetic nanoparticles of BaFe were added with a concentration of 1 g/l. BaFe nanoparticles with purity 99.5% and a nominal dimension of 5 nm were purchased from Sigma-Aldrich Company. The electrodeposition process into the nanopores of the AAO membrane was conducted in the galvanostatic conditions at a current density of 1 A/dm² and deposition time ranged from 1 to 2 hours. The volume of the electrodeposition solution was of 250 ml and maintained at temperature of $50 \pm 2^\circ\text{C}$.

3. RESULTS AND DISCUSSIONS

The obtained template membrane was investigated by SEM technique. Fig. 2 presents a general top view of the AAO template backside surface after a first-step anodization process. The morphology is characterized by a hexagonal ordered array of compact packing cells formed from nanopores of the same dimension [13]. The AAO membrane had thickness of about 20 μm , the inter-nanopores distance was 50 nm and the nanopore surface density was about 1000 cm⁻².

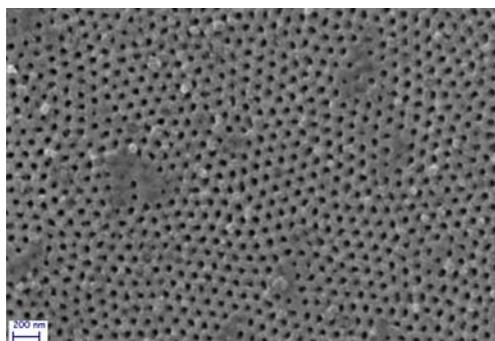


Fig. 2 – SEM image of the AAO template surface after a first-step anodization process.

The SEM images presented in Figs. 3a) and b) show a detail and a cross section of AAO template respectively, after two-step anodization process evincing a columnar structure of nanopores in perpendicular direction to the surface.

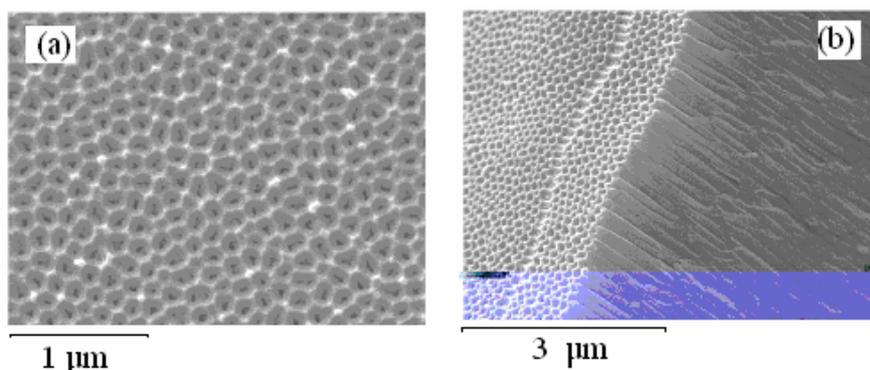


Fig. 3 – Detailed SEM images of AAO template: a) top surface; b) cross section after two-step anodization process.

The arrangement of the columns follows fairly the hexagonal cells structure. This effect was highlighted by comparing the morphology of the oxide on the surface with the inner surface of AAO template.

The galvanostatic curves for the Ni-Co and Ni-Co/BaFe nanowires, during the electrodeposition process into AAO template, have been recorded (Fig. 4). The nanowire nucleation in the nanopore of nanocell in the presence of nanoparticles was more difficult to observe. After 1 h deposition, the nanowires were well-defined with a length of about 10 μm, for both cases. The Ni-Co and Ni-Co/BaFe nanowires show high morphological and structural quality (Figs. 5a and b).

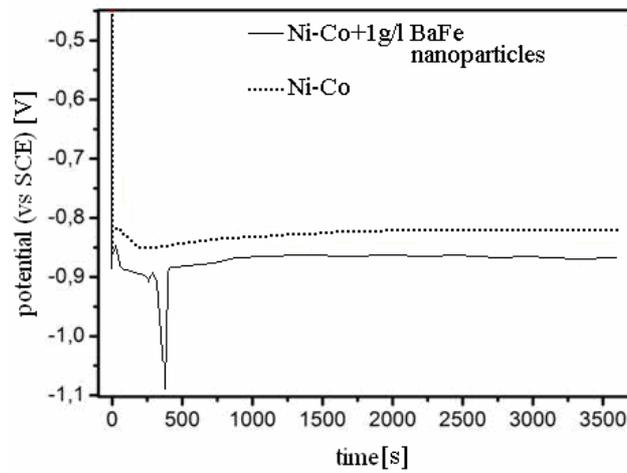


Fig. 4 – Variation of potential vs. time during galvanostatic cathodic deposition of Ni-Co (···) and Ni-Co/BaFe (—) nanowires.

In order to modify the magnetic properties of the Ni-Co nanowires, it was tried the possibility to electrodeposite the nanocomposite nanowires consisting of Ni-Co magnetic matrix and BaFe (magnetic) nanoparticles. For a good dispersion of the nanoparticles, before the electrodeposition process, the electrolyte that contained BaFe nanoparticles was treated with ultrasounds for 15 min.

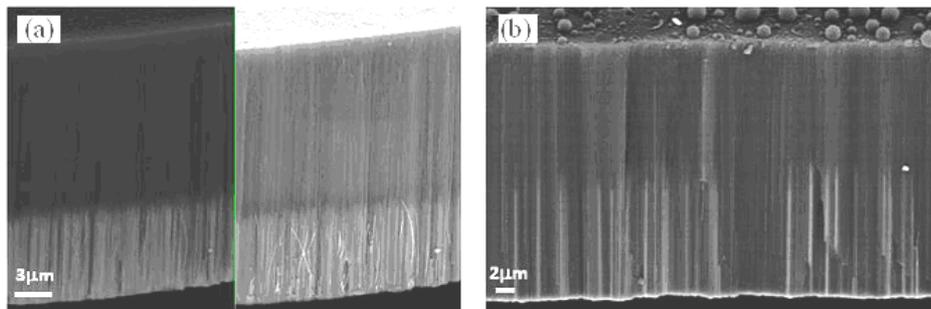


Fig. 5 – a) SEM images of Ni-Co; b) Ni-Co/BaFe magnetic nanowires.

For both cases, the compositional analysis performed with EDAX equipment (Quanta 200-FEI) indicated the formation of the rich-nickel nanowires and a significant proportion of ferrite into the structures formed from the solution that contained nanoparticles. The average composition of Ni-Co nanowires is shown in Fig. 6 when the electrolyte is without nanoparticles.

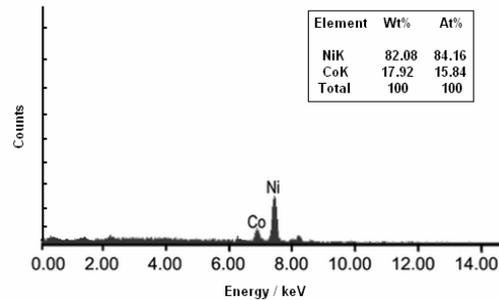


Fig. 6 – EDAX spectrum of the Ni-Co alloy nanowires within AAO template pores.

The XRD experiments were carried out using Philips PW 3020 equipment with PW 1830 HT generator. XRD patterns showed that the obtained nanowires had a crystalline structure (Fig. 7a – 2θ ranging from 40° to 46° ; Fig. 7b – 2θ ranging from 75° to 97°). The X-ray analysis of the deposits shows the presence of Ni and Co with a structure progressively changing with their chemical composition depending on the ferrite nanoparticles incorporation.

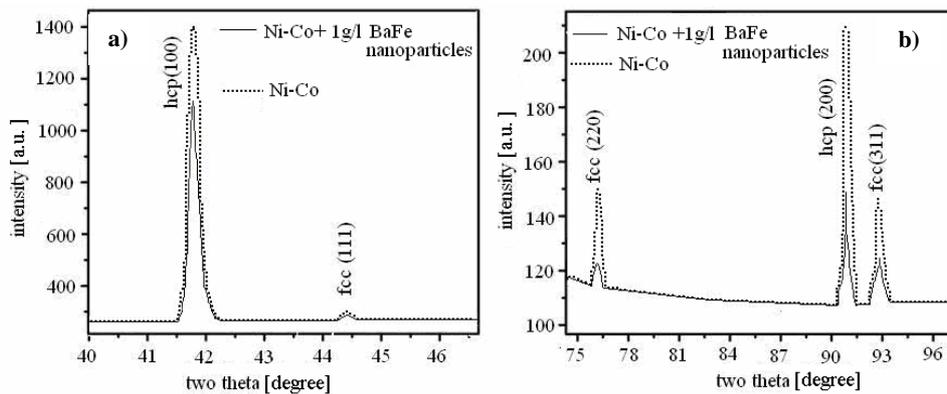


Fig. 7 – Fragments of X-ray patterns for Ni-Co (···) and Ni-Co/BaFe (—) nanowires.

The obtained nanowires from Ni and Co sulfamate electrolytes have shown the formation of a mixture of hcp and fcc crystal structures and obviously are polycrystalline [13]. Ni-Co nanowires showed hcp-Co phase textured in the [100] and [200] crystallographic directions, and fcc-Ni phase. The amount of BaFe from the nanowires being very small it cannot be clearly detected in XRD spectra. A similar behavior was observed for Ni-Co/BaFe nanowires showing an influence of the BaFe nanoparticles from the electrolyte bath on the nucleation process and on the structure growth. The diffraction peaks obtained at $2\theta = 44.4^\circ$, 76.5° , and 92.8° (curve (···) of Figs. 7a and 7b) can be assigned to the characteristic (111), (220),

and (311) planes of fcc structure of Ni (JCPDS, 04–0850). The typical diffraction peaks can prove that the Ni-Co alloy was synthesized. A similar behavior was observed during BaFe nanoparticles incorporation in the nanocomposite nanowires. The effect consists in diminishing of crystallization process, decreasing the crystallite size and low phase contents. Therefore, a weak intensity of peaks could be observed.

EDAX results of Ni and Co contents have a local character and XRD data having a regional feature show the presence of certain texture degree of Co due to the BaFe nanoparticles incorporation.

A characterization of the magnetic properties was made by using a MPMS XL magnetometer (Superconducting Quantum Interference Device – SQUID) that works at room temperature, in helium atmosphere. It allowed monitoring very small changes in magnetic flux and so discovers the magnetic properties of samples. The magnetization vs. magnetic field curves of the membrane containing nanowires was recorded by magnetic field applied parallel or perpendicular on AAO template.

In order to univocally ensure the ferrite incorporation in the Ni-Co matrix a high resolution electron microscopy analysis was necessary to have more detailed information about the structure. In this aim, membrane was partially removed and nanowires could be studied separately. The electron microscopy highlighted that the nanowires have been released without damaging and the nanoferrite particles distribution was clearly observed having size smaller than 10 nm. A good distribution of the nanoparticles in Ni-Co deposit was obtained and a poor agglomeration was detected proving the formation of nanocomposite nanowires. The EDAX analysis confirmed the presence of the BaFe nanoparticles and Ni-Co alloy matrix. The incorporation of the BaFe nanoparticles in Ni-Co nanowires induced a substantial change in the magnetic response of the Ni-Co nanowires (Figs. 8a and b).

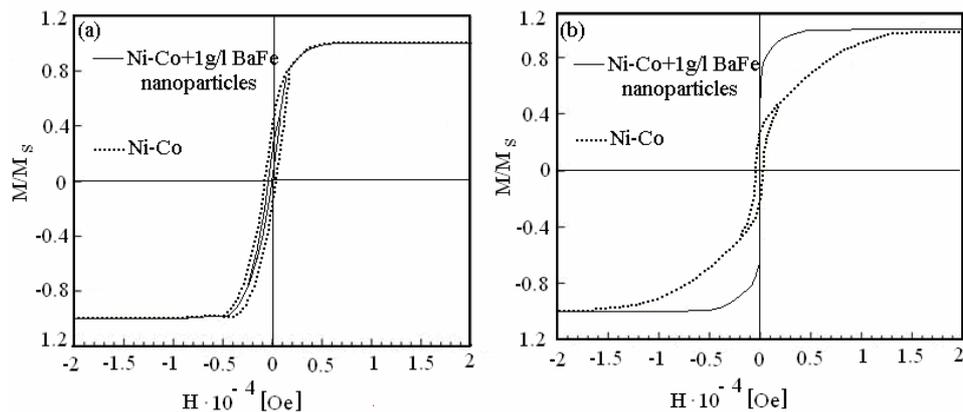


Fig. 8 – Relative magnetization vs. applied magnetic field (a) parallel and (b) perpendicular to AAO membrane surface.

The magnetization curves presented in Fig. 8 show that the BaFe nanoparticles have a weak influence on the magnetic behavior of the nanowires when the applied magnetic field is perpendicular to the membrane surface (Fig. 8b). Namely, the square shape of the loop and widening is prominent when the magnetic field was applied parallel to the nanowire axis (Fig. 8b) compared to the perpendicular direction. Therefore, the easy axis of magnetization is along Ni-Co nanowires while hard axis is normal to the nanowires. This behavior is associated with increasing of magnetocrystalline anisotropy for the nanocomposite nanowires and can be used in some practical applications. The ferrite nanoparticles incorporated in nanocomposite nanowires lead to an increasing of magnetic remanence when the membrane is perpendicular to the magnetic field. This behaviour can be connected with the agility orientation of total magnetic moment of atoms in the external magnetic field.

4. CONCLUSIONS

The self-ordered AAO membranes have been fabricated and used as template in order to grow Ni-Co and Ni-Co/BaFe nanowires. Afterwards, those were characterized and analyzed by SEM, EDAX and XRD techniques showing high-purity nanowires no oxides being identified. Furthermore, the growth directions were preferential along [111] crystalline direction for Ni and along [220] crystalline direction for Co. These were attributed to attempt to increase the nanowires into the nanoporous AAO membranes.

The narrow width of AAO pore diameters and the electrodeposition parameters could influence the polycrystalline character of the studied metallic nanowires.

The presence of BaFe nanoparticles has a strong influence on the magnetic behavior of the composite nanowires when the magnetic field is applied parallel to the AAO membrane surface (perpendicular to nanowire axis). Ni-Co alloy nanowires are easily magnetized in the direction perpendicular to the nanowire axis, but are difficult to magnetize in the parallel direction. The results show that it is possible to obtain metallic nanocomposite structures at nanometric scale through the incorporating nanoparticles by electrochemical method. Nanowires prepared by electrocodeposition process into AAO template containing magnetic nanoparticles exhibit different properties both from those without nanoparticles and from bulk nanocomposites.

The composition of the electrolytic solution and the selected electrodeposition conditions, aggregation of the nanoparticles and their sequential incorporation in the porous templates open new perspectives in the field of magnetic nanostructures with modular properties.

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