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Morphology, structure and magnetic study of permalloy films electroplated on silicon nanowires

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ABSTRACT

We report the effect of deposition potential on the morphology, structure and magnetic properties of $Ni_{80}Fe_{20}$ (Permalloy: Py) deposits, elaborated by electrochemical process onto silicon nanowires (SiNWs). The morphology of SiNWs and Py/SiNWs were performed with scanning electron microscopy (SEM). The SEM micrographs reveal the formation of SiNWs and clearly show a change in the morphology with the deposition potential. The analysis of X-ray diffraction spectra shows a change in the texture with the deposition potential. The grain size, the lattice parameter and the strain were studied as a function of the deposition potentials. From hysteresis loops, we have shown that the magnetization easy axis is the plane of the samples.

1. Introduction

In the recent decades, nanomaterials science and nanotechnology became one of the most attractive areas for scientists, both at fundamental and technological level, because of the wide range of possible applications. The nanowires are considered as part of nanomaterials which have potential applications in the field of optics, electronic components, electronic batteries connectors, solar cells and magnetic recording media [1–3]. Permalloy ($Ni_{80}Fe_{20}$) is one of the most used materials in the field of magnetism due to its interesting properties such as high permeability, low coercivity and low magnetic anisotropy. In the last years, scientists have devoted their interest to the study of NiFe nanostructures using nonostructured templates like nanopores. Among these studies, we can cite the recent work of A. Llavona et al. [4] who used a nanoporous membrane as a template to elaborate NiFe nanowires and studied the enhancement of anomalous co-deposition of the NiFe alloy. X. Zhang et al. [5] used anodized aluminum oxide templates to synthesize FeNi nanowires and nanotubes by electrochemical process. The authors found that the nanowires present a (111) preferential orientation and conclude that the magnetic properties depend strongly on the length of FeNi alloy nanowires and nanotubes. R. Vasic et al. [6] have prepared NiFe nanowires on Si nanopores by electrochemical etching; thereafter, they studied the dielectric relaxation of the nanostructure in high magnetic fields. S. Ouir et al. [7] studied the growth of Permalloy in the macroporous and mesoporous silicon and the effect of this microstructure on the composition and the magnetic properties of the NiFe deposits. The present investigation is motivated by the lack, as far as we know, of a detailed study dealing with electrodeposed Py. In this perspective and in order to develop a new Permalloy nanostructure with interesting properties, we have developed this research work about the synthesis of NiFe deposits on Si nanowires. The effect of electrodeposition potentials on the microstructural, structural and magnetic properties will be studied.

The SiNWs were elaborated using metal assisted chemical etching method; the details of their formation are given in Sections 2.·1. Details about electrochemical deposition of NiFe films on SiNWs are also given in Section 2.1. The different characterization methods used in this study are exposed in Sections 2.·2. The morphological, structural and magnetic properties were investigated with Scanning Electron Microscopy (SEM), X-Ray

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diffraction (XRD) and Vibrating Sample Magnetometer (VSM). In Section 3, the obtained results and their discussions are developed. A conclusion is given in Section 4.

2. Experimental methods

2.1. Elaboration of SiNWs and NiFe deposits

We have used an n-type Si (100) to prepare three samples of silicon nanowires, SiNWs, using a metal assisted chemical etching method. Before etching, the Si wafers (substrates) were cleaned in baths of trichloroethylene, acetone and ethanol using an ultrasonic container. Next, the cleaned wafers were immersed in 10% HF aqueous solution for 5 min at room temperature in order to remove the native oxide. Then, these samples were dipped for 1 min into an $AgNO_3$/HF solution used to electroless deposition of Ag nanoparticles (AgNPs) at room temperature. Subsequently, the Si samples coated with AgNPs were immersed, for 1 h, into H_2O_2/HF solution to perform a chemical etching at room temperature. Finally, the etched Si samples were soaked in a solution of HNO_3 (69%) to remove the residual of AgNPs, then cleaned with de-ionized water and dried under nitrogen.

The obtained SiNWs were used as substrates to deposit Permalloy with electrodeposition method using a multi-potentiostat VMP3. The electrolyte used is composed of 0.5 M $NiSO_4$, 0.02 M $FeSO_4$, 0.4 M H_3BO_3 and 0.012 M saccharin, at ph = 3 adjusted by H_2SO_4 solution. All chemicals elements were of analytical grade and they were used without further purification. Then, they were mixed in deionized water. To minimize the oxidation reactions, the solutions were freshly prepared each time before plating. The deposition was carried out at room temperature, in a three electrode cell. A platinum mesh was used as counter electrode and an Ag/AgCl electrode as reference electrode. Different potentials varying from −1.6 V/(Ag/AgCl) to −2 V/(Ag/AgCl) were applied to deposit Permalloy films on the SiNWs.

2.2. Characterization methods

Cyclic voltammetry (CV) was used to define potential regions and to characterization the nickel and iron deposition process on SiNWs. The morphology of the samples was investigated by Philips XL 30 scanning electron microscope. The observations were performed on the surface and for a cross section of samples under different magnifications. The chemical composition was performed with Energy Dispersive X-ray Fluorescence Spectroscopy EDXRF. To study the evolution of the structure, X-ray diffraction experiments (XRD) were performed with a Philips X-pert diffractometer using Cu Kα radiation (1.54056 Å) with 2θ ranging from 40° to 90°. The hysteresis loops were obtained at room temperature using a vibrating sample magnetometer (VSM), with an external magnetic field H applied parallel and perpendicular to the samples surface.

3. Results and discussions

3.1. Cyclic voltammetry

Typical cyclic voltammogram for deposition of NiFe alloy is shown in Fig. 1. The cycle exhibits two cathodic peaks labeled P1 and P2 and anodic peaks. The peak P1 around −1.2 V, corresponds to the NiFe deposition. The second peak P2, at approximately −2.7 V is attributed to H_2 emission. The anodic peak observed at positive potentials (around 1.1 V) corresponds to the oxidation which induces dissolution of Py deposit [8].

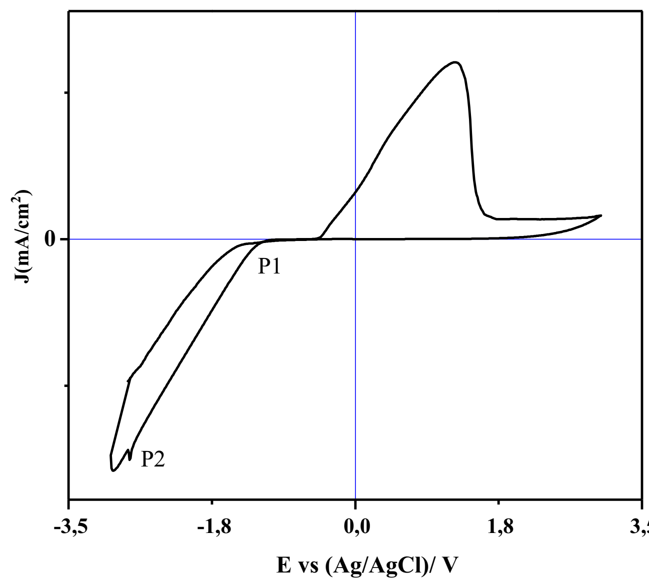

Fig. 1. Cyclic voltammetry scan for silicon nanowires in NiFe electrolyte.

3.2. Morphology and EDXRF analysis

The morphology of silicon nanowires was observed by SEM. The micrographs of the sample surface for different magnifications are shown in Fig. 2. We observed that the etching was done uniformly on the silicon surface resulting in the formation of nanowires with grouped tips forming bouquets. The formation of these bouquets is attributed to the van der Waals attraction [9]. According to these images; the dimensions of nanowires are about some micrometers in length and ten nanometers in diameter. For a better observation of silicon nanowires, we performed a cross-section of the samples; the images of the SiNWs for different magnifications are shown in Fig. 2(c and d). We observed a large amount of silicon nanowires well aligned and perpendicular to the surface. The length of the SiNWs was estimated to be approximately 25 μm.

SEM observations were also performed on Py deposited on SiNWs. The micrographs of samples deposited at voltages of −1.6 V, −1.8 V and −2 V (Ag/AgCl) are shown in Fig. 3a, b, and c respectively. For a potential of −1.6 V, Fig. 3(a), we observe a non-uniform deposition of Py on the ends of nanowires. Indeed, we see the formation of small agglomerated particles with various forms. For −1.8 V (see Fig. 3(b)), the non-uniform deposition is still observed and no change in the morphology can be evidenced. For the sample deposited at a voltage of −2 V (Fig. 3(c)), a change in the morphology of the deposits is clearly observed. Indeed, we note the apparition of spherical NiFe particles on SiNWs. The diameter of these particles is estimated to be about 2 μm. From these SEM micrographs, we evidenced that the Py deposits grow on SiNWs tips. Moreover, we showed that the applied voltage affects drastically the morphology of NiFe samples deposited on SiNWs.

Energy-dispersive X-ray fluorescence (EDXRF) analysis was made on all NiFe/SiNWs deposits. An example for the sample deposited at −2 V is shown in Fig. 4. From this spectrum we observe that only the peaks corresponding to Fe and Ni elements are present. The qualitative analysis gives a composition of NiFe deposits which is close to that of Permalloy (80%Ni:20%Fe).

3.3. Structure

X-ray diffraction experiments have been performed on all samples. The XRD spectra of Py/SiNWs deposits corresponding to different applied potentials are presented in Fig.5. For the samples