Electrodeposited nickel nanowires for magnetic-field effect transistor (MagFET)

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ABSTRACT

The growing interest in magnetic nanowires (NWs) is connected to possibility of employing them for advanced applications in wide technological fields, such as data storage and biotechnology. In addition, NWs can be used as sensor devices for several applications, since they present high sensitivity to their environment. One of the major challenges when dealing with transport measurements in NWs is to trap them between electrodes, which allows electrical characterization and therefore fabrication of nanowire-based devices. Electrically neutral NWs can be deposited by dielectrophoresis (DEP) method, which requires the application of an alternating electric field between electrodes. In this work, Ni nanowires (NiNWs) fabricated by electrodeposition technique and properly dispersed in a dimethylformalmide (DMF) solution were deposited on top of Pt electrodes using the DEP method. The deposited NiNWs exhibit initially a Schottky-like current versus voltage behavior due to the high contact resistance between NiNW and electrode. Its reduction down to three orders of magnitude, reaching value less than the NiNW resistance, was achieved by depositing an ion beam-assisted 10 nm-thick Pt layer over the NWs extremities. Therefore, this method presents a suitable process of NWs deposition and electrical characterization. This can be used for investigation of electrical transport properties of individual NWs, one can use the present method for fabrication of magnetic field-effect transistors (MagFET).

Index Terms: nickel nanowires; nanowire-based devices; magnetoresistance, Focused Ion Beam (FIB).

I. INTRODUCTION

Nanowires-based devices have been implemented due to their ultra-low power consumption and high sensitivity to electrical, physical or chemical stimuli [1-5]. One of the biggest challenges to fabricate and study electrical transport properties of NWs-based devices, such as semiconductor- and carbon-based transistors, is the appropriate manipulation of the NW towards electrodes [6,7]. Several techniques, such as focused ion beam (FIB) and atomic force microscopy (AFM) manipulation have been used for fabrication of NWs-based devices [5]. Alternatively, metallic NWs suspended in a dielectric liquid medium can be directly manipulated through an external applied electric field [1,5,8-13]. When a neutral NW is placed inside a non-uniform electric field region, the electric charges are redistributed within the NW and in the liquid portion of the liquid-solid interface, building up an induced electric dipole moment [14]. Since the Coulomb forces on either sides of the electrical dipole moment can be different, a net force is exerted on the NW, which is known as the dielectrophoretic force. The force direction depends on the relative polarizabilities of the NW and the diluting medium, inducing the former to move towards or against the region of higher electric field intensity. Such motion is called dielectrophoresis (DEP) [6-14]. DEP manipulation can be controlled by varying the frequency and magnitude of the applied electric field [14]. Also, a large electrode array can be defined by lithography such that DEP can take place in a large number of electrodes, which increases the process throughput. In addition, DEP allows high selectivity and sensitivity analysis, since it is directly dependent on the dielectric properties of the particles and diluting medium [14].

Therefore, in this work, properly isolated NiNWs, with length of around 4 μ m and 35 nm of diameter, obtained by electrodeposition into pores of anodized alumina membrane [15], were dispersed

in a dimethylformalmide (DMF) solution and dielectrophoretically manipulated to make electrical contact between electrodes. Optimized electrodes geometry and DEP electrical parameters [13] were taken for NiNWs deposition. In addition, electrical characterizations of the NWs and of the contact resistance between the NWs and electrode were performed by current versus voltage curves. Significant reduction of contact resistance was achieved by ion beam assisted deposition of Pt cap layers on the NW extremities.

The main objective of this work is to study Ni nanowires for future MagFET applications. These devices are using the anisotropic magnetoresistance (AMR) phenomenon, a quantum-mechanical effect based on spin-orbit coupling, that allows current modulation through the NW by an external magnetic field [16]. Highly anisotropic structures, such as NWs (aspect ratio higher than 50), present mainly shape-anisotropy magnetoresistance (MR). Other parameters, such as tensile stress and crystallographic configuration, can contribute to MR behavior of nanoparticles through magneto-elastic and magneto-crystalline anisotropies, respectively [16,17].

II. EXPERIMENTAL DETAILS

A. Nanoporous alumina membranes

For the growth of nanowires, nanoporous alumina templates, obtained by a standard two-step anodization method of high-purity (>99.997%) aluminum foils, were employed [15,18]. Prior to anodization, samples were properly cleaned and degreased. For surface improvement, samples were also electropolished at 20 V for 2 min in an ethanol:perchloric acid (4:1) solution. With this procedure, a mirror surface was obtained, setting the necessary conditions for a well-organized porous structure to be obtained. Anodizations were performed at a constant voltage of 40 V for 15 h, with a 0.3 M oxalic acid solution (used as electrolyte) at a temperature between 2 and 6 °C. These anodization conditions resulted in nanoporous alumina substrates with an average pore diameter of approximately 35 nm and separation around 100 nm. To be able to electrodeposit metallic nanowires in the nanoporous alumina templates without having to manipulate the thin and fragile membranes, the deposition current has to flow through the oxide barrier, which therefore must be thinned down. The reduction procedure gives origin to dendrites (small channels in the alumina barrier-layer) enabling the current to tunnel through the resulting barrier [19,20].

B. Nanowires growth

A pulsed electrodeposition method was used to grow NiNWs [19,21]. This method combines a constant current pulse for material deposition, followed by a constant potential pulse for both discharging the barrier-layer and repair membrane cracks. Finally, a resting time with no potential or current applied, enables the recovery of the ion concentration at the bottom of the pores. The electrolyte employed for Ni deposition was a standard Watts bath (NiSO₄ · 6H₂O, NiCl₂ · 6H₂O and H₃BO₃). Deposition pulses with a current density of 38 mA/cm² were taken. The electrodepositions were carried out at 30 °C [20]. The resulting NiNW were $4 \pm 1 \mu$ m-long with $35 \pm 5 n$ m of diameter. They present ferromagnetic properties with almost isotropic behavior and the easy axis along the longitudinal nanowire direction [20].

C. Device fabrication

A 300 nm-thick SiO₂ layer was grown on a n⁺ – type Si (100) wafer (electrical resistivity of 1~10 Ω .cm) by wet thermal oxidation in a conventional furnace (Fig. 1a). Then, photolithography was performed to define the electrodes region. 80 nm-thick Pt layer was sputtered by a physical vapor deposition (PVD) system and lift-off process was carried out to define electrodes (Fig. 1b).

The fabricated NiNWs were released from the membrane by chemical etching with a 1 M NaOH solution at 27 °C under agitation. They were then cleaned with deionized water (18 MΩ.cm) and dispersed in DMF, in order to avoid NWs clusters formation. The NiNWs deposition was performed by DEP with optimized set of parameters [13], conducted with a HP 8116A Pulse/Function Generator configured with 6 V_{pp} and null offset (Fig. 1c). The sinusoidal signal was generated for a frequency range between 50 kHz to 1 MHz. Before DEP process, the solution (concentration of 10⁸ NiNW. mL⁻¹) was sonicated for 120 seconds, in order to uniformly disperse the NiNWs into the DMF. For each pair of electrodes, the DEP field was applied during 60 seconds on a solution volume of 1μ L. The DMF excess was rinsed with deionized water (18 M Ω .cm) before being dried with N₂. Finally, a 10 nm-thick cap layer of Pt was deposited on the NiNWs extremities to reduce the contact resistance with the electrodes (Fig. 1d), using a Ga+ focused ion beam (GaFIB)/scanning electron microscope (SEM) with a gas injection system (GIS) tool [22]. The GIS is an available feature with the Ga⁺ FIB/ SEM dual beam system that allows the deposition, using ion or electron beams, of metallic materials, such as Pt [13,22-24]. The ion beam source used in



Figure 1. Schematics of device fabrication: (a) dielectric layer formation on top of n⁺-Si wafer by thermal oxidation; (b) electrodes definition by photolithography and lift-off; (c) NiNW deposition on electrodes by DEP experiment and (d) contact resistance reduction after the deposition of Pt layer by GIS-FIB. Adapted from [13]. Copyright 2015 by American Vacuum Society. Adapted with permission.

this work was gallium ions (Ga⁺) from a FEI Nova 200 Nanolab GaFIB/SEM dual beam system with energy of 30 keV, current of 10 pA and a null tilt angle. Under these conditions, the milling process and damage of NiNW and electrodes were significantly reduced [13,25].

The precursor gases are introduced close to the sample by the GIS and adsorbed on the substrate surface. Secondary electrons, with energy in the range of $2 \sim 80$ eV, are produced in the scanned region of the substrate by interaction with ion or electron beam, which cracks precursor molecules over the defined area [23,26]. Volatile components of the process then leave the surface and are pumped away by the vacuum system. The precursor gas of Pt is a platinum-based organometallic compound ((CH₃)₃PtCpCH₃) [23,24,26].

III. RESULTS AND DISCUSSION

The electrodeposited NiNWs present a polycrystalline structure with grain of average size 20 \pm 8 nm, as shown in Fig. 2. Both Electron Energy Loss Spectroscopy (EELS) and Energy Dispersive Spectroscopy (EDS) analyses were taken to confirm and map NiNW composition. EELS results exhibit a Ni core surrounded by a 4 ± 1 nm-thick native oxide (Fig. 3a). The EDS spectrum confirmed NiNW composition with Ni La and O Ka transition energies over the inspected region (Si peak refers to the substrate) (Fig. 3b). Visual inspection of the gap region by SEM (Figs. 4a and 4b) presents NiNWs deposited on Pt electrodes after DEP experiment. For optimized DEP parameters (3 V_{PP}, 100 kHz, null offset, 60 s, rectangular electrodes) the efficiency of NiNWs deposition was up to 85%, with only a few NiNWs deposited each time, which allows device fabrication [13]. The total electric field distribution over the gap area was simulated using COMSOL Multiphysics simulation tool (Fig. 4c), showing high intensity and good uniformity over the gap region.



Figure 2. Transmission Electron Microscopy (TEM) images of NiNW: (a) top view; (b) crystallographic planes of the polycrystalline structure and (c) dark-field image showing size distribution of planes under the same diffraction condition.



Figure 3. (a) EELS analysis for composition mapping of the NiNW and (b) EDS spectrum taken to confirm composition of the NiNW deposited on the top of Si substrate. Si K α (1740 eV), O K α (525 eV) and Ni L α (852 eV) edges are present.



Figure 4. SEM analysis of NiNW deposited on Pt electrodes after DEP experiment for $V_{PP} = 3$ V and frequency of (a) 100 kHz and (b) 600 kHz. (c) Static simulation (\pm 3 V between electrodes) using COMSOL of the total electric field intensity distribution over the gap area, showing good homogeneity over the gap region (20 µm-long line) as shown in (d). Adapted from [13]. Copyright 2015 by American Vacuum Society. Adapted with permission.

As demonstrated, DEP is an adequate tool to insert NWs between electrodes for electrical transport measurements [13]. However, when the nanowire touches the electrodes (Fig. 5a), a large contact resistance is usually present, leading to a Schottky-like behavior (non-linear). After depositing a 10 nm-thick cap layer of Pt on the NiNWs extremities (Fig. 5b) to reduce the contact resistance, the resulting behavior is ohmic (linear) [13,22].

The current-voltage (I-V) measurements were obtained with a standard four-probe measurement with a Keithley 2400 source by applying current (without exceeding 6 μ A to avoid NiNWs damage due to heat dissipation) while measuring voltage with a four-wire setup. We observed a transformation from a non-linear



Figure 5. SEM of NiNW (a) as-deposited between electrodes and (b) after Pt deposition by GaFIB and GIS tool for reduction of contact resistance. Adapted from [13]. Copyright 2015 by American Vacuum Society. Adapted with permission.

behavior for as-deposited system to a linear one, after Pt layer deposition, as well as a resistance reduction (Fig. 6a). From the obtained NiNW resistance $(3 \text{ k}\Omega)$ after subtracting the electrodes resistance contribution (120 Ω), we obtained a resistivity value for NiNW $(\rho_{NiNW} = 54 \ \mu\Omega.cm)$ that is consistent with those of similar dimensions [27]. Figure 6b presents the parallel-equivalent resistance as a function of the number of deposited NWs, both in logarithmic scale, before and after Pt deposition. The linear fit slopes (-1.2 \pm 0.2 Ω .NiNW⁻¹ for as-deposited NiNWs) and (-1.0 ± 0.1 Ω.NiNW⁻¹ after Pt deposition) are in agreement with the ideal case (-1 Ω .NiNW⁻¹), which confirms the parallel-equivalent resistance law for the NiNWs. In addition, the offset between the linear fits indicates the resistance reduction by the Pt deposition method using Ga+ FIB/SEM and GIS. Reference [13] evaluates the effects of Ga+ implantation on NiNWs for Pt deposition. The ionic bombardment on the NiNW/Pt electrodes structure yields to an amorphization depth



Figure 6. (a) I x V curves for NiNW before (circle, left and down axes) and after (line, right and up axes) 10 nm-thick Pt layer deposition by GIS tool of GaFIB. (b) Equivalent parallel resistance versus number of parallel deposited NiNW before and after Pt deposition. Adapted from [13]. Copyright 2015 by American Vacuum Society. Adapted with permission.

of around 25 nm in the region of Pt deposition. Thus, it remains a thickness of at least 10 nm of polycrystalline NiNW not reached by Ga+ ions, i.e. the damage created is not over the entire NW diameter. The resistance of the resulting structure increases around one order of magnitude to this effect. Nevertheless, the Pt deposition was limited to the NWs very extremities to prevent increase of their resistance by Ga amorphization [13].

The electric resistivity of one single NiNW, $\rho_{\rm s}$ was measured as a function of temperature, T, using a standard four-probe technique in a Physical Property Measurement System (PPMS), in the range of 2 - 300K (Fig. 7), showing metallic behavior, as expected. No Pt cap layer defined by FIB was necessary in this measurement, given the low contact resistance present between the NiNW and the Pt electrodes. The residual resistance of the NiNW is $\rho_0 = 27 \ \mu\Omega.$ cm, which is in good agreement with similar dimensions NiNW [4,18]. The relative ratio of resistivity ρ (300 K) / ρ (4.2 K) = 2 is much smaller than the value of 47 for the bulk [4]. Since the NW dimensions and grain-size are similar to the mean free path of Ni (~ 10 nm), the increase in resistivity can be attributed mainly to the grain-boundary scattering [16,17].

The variation of ρ as a function of the magnetic field, *H*, was measured at 300 K for one isolated nanowire with the current flowing perpendicular to the applied field. The magnetoresistance signal, defined as:

MR (*H*) =
$$[\rho (H) - \rho_{max}] / \rho_{max}] \ge 100,$$
 (1)

where ρ_{max} is the maximum resistivity value, decreases down to around -1% as the magnetic field increases (inset of Fig. 7), as expected for NiNWs [20,28]. This



Figure 7. ρ x T curve of one single NiNW measured with a PPMS system, showing metallic behavior and a residual resistance of 27 μ Ω.cm at 2K. Inset: magnetoresistance measurement of one single NiNW at 300 K.

behavior is attributed to the quantum-mechanical effect of spin-orbit coupling for magnetic fields below 10 kOe [20,28].

IV. SUMMARY AND CONCLUSIONS

This work presented DEP manipulation of polycrystalline NiNWs over Pt electrodes defined by photolithography and lift-off. Adequate individual NiNW electrical measurements are allowed by the successful contact resistance reduction through deposition of 10 nm-thick Pt cap layer on the NW extremities by GIS-GaFIB. From current versus voltage measurements a consistent value of resistivity was obtained. Ion bombardment by GaFIB increases NiNW resistance due to Ga⁺ ion damage albeit does not prohibit the process adequacy. Moreover, the studied NiNWs are a promising feature to be used as sensors devices, since they can be manipulated with high efficiency to make contact with electrodes and their electrical, thermal and/or optical output signals (in response to the environment stimulus) can be further processed [28]. In addition, NiNWs present ferromagnetic properties, which allow their low current levels to be controlled through magnetic fields, like a MagFET device. These magneto-transport properties can be thought as a promising alternative to the traditional Si-based MOSFET devices.

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