

Ni NANOWIRES ELECTRODEPOSITED IN SINGLE ION TRACK TEMPLATES

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30- μm thick polycarbonate foils were irradiated with single swift heavy ions (e.g. Au 11.4 MeV/nucleon). After etching with solutions containing NaOH and methanol, templates containing single conical pores were obtained. The size and the shape of the nanopores depend on etching conditions such as time of etching, concentration and temperature of the etching solution. By electrochemical deposition of nickel, nanowires were grown in the single pores. The nanowires were contacted by sputtering a gold layer on top of the membrane. The magnetic measurements indicated that the nanowires possess around 1% anisotropic magnetoresistance. The current-voltage characteristic of a Ni nanowire shows a linear behavior for current densities smaller than 10^8 A/cm^2 . The maximum current density that a single Ni wire can withstand was found to be $3 \times 10^8 \text{ A/cm}^2$.

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1. Introduction

Magnetic nanowires represent an important family of magnetic structures, which during the last few years attracted an increased interest arising from unique physical (e.g. electronic, optical, magnetic, mechanical) properties on account of their very restricted size. In addition, their numerous potential applications in a variety of fields, such as physics, chemistry, materials and biosciences, help promote investigations on these novel nanostructures.

Among the methods used in order to obtain nanowires, the filling of channels in a template is a straightforward approach. This technique consists in synthesizing the desired wires within the pores of a membrane [1,2]. Ion track membranes provide significant advantages over other usable templates, particularly in view of the degree of freedom they offer to produce nanowires of a specific number and with different compositions. The host template can be made from a range of materials including polymers and glasses, the most widely used being polycarbonate, polyimide and polyethylene. The method of producing nanowires by means of ion track membranes consists of several steps: (1) irradiation of a polymer film with swift heavy ions in order to create long, narrow and absolutely straight damage trails along the ion trajectories; (2) conversion of these so-called latent tracks into thin pores by chemical etching; (3) filling of pores with a large variety of materials [3,4,5].

Recent progress on magnetism and magnetic materials has made the magnetic nanowires a particularly interesting class of objects for both scientific and technological applications. For example, research on subjects such as giant magnetoresistance [2, 6], anisotropic magnetoresistance

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[6,7,8] and current-induced magnetization switching [9] has opened up the possibility of utilizing the nanowires for information data storage or as magnetic sensors.

In order to measure the magnetoresistance of nanowires, one single wire needs to be contacted. Several methods have been developed recently for this purpose. One method requires the template technique combined with electron beam lithography, thus being restricted to relatively few laboratories [10]. Another method contacts the fastest growing wire from a multi-porous membrane by a "floating electrode" [11]. A third method localizes and contacts randomly distributed [12] or flow-aligned [13] nanowires on a substrate by photo- or electron-lithography.

In this report we present a new method for preparing and contacting single Ni nanowires. This method circumvents the necessity of uneven growth rates of nanowires, required by the floating electrode technique, a wider range of growth conditions for nanowires becoming thus possible. Also, no manipulation of the nanowire or lithographic techniques are needed to contact the nanowire, making the method very simple and accessible to a large number of laboratories.

2. Experiment

Polymer foils (Makrofol N, Bayer Leverkusen) of circular shape (thickness 30 μm , diameter 4 cm) are irradiated at the UNILAC (UNIversal Linear Accelerator) of GSI (Darmstadt, Germany) with single swift heavy ions through a circular aperture with a diameter of 200 μm in a metal mask of 500 μm thickness. The ion fluxes are in the range 10^3 - 10^4 ions $\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$, so that the probability of double hits is very low. As soon as a detector located behind the sample has recorded one ion hit, the ion beam is blocked by a fast chopper system and the next sample can be set for irradiation.

For better manipulation, a thin gold layer of approximately 0.2 μm thickness is sputtered on the rough side of the irradiated foil, which is used as a cathode during track etching and growth of the wire. We observed during our previous experiments that, due to the flexibility of the polymer foil, the nanowires can easily break while handling the sample. In order to provide a better mechanical stability of the sample, the gold layer is additionally reinforced by inserting the sample in a cell and electrodepositing a copper layer (10 μm thick). To increase the mechanical stability even further, the polymer foil is taken out of the cell, mounted on a printed circuit board by a double-adhesive copper tape and then reinserted (Fig. 1).

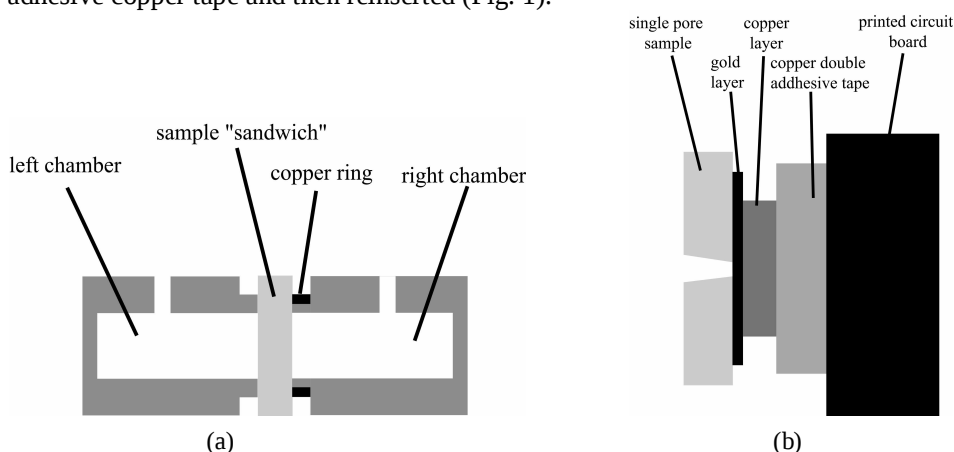


Fig. 1. (a) Cell used for single-wire deposition; (b) sample "sandwich".

Track etching is performed in the left cell chamber using a solution of 5M NaOH with 50 vol. % methanol at 50°C. To monitor the etching process and identify the exact moment when the pore is completely etched through, a gold electrode is immersed in the etching solution, and a pulsed square-shaped voltage with a frequency of 0.1 Hz and an amplitude of 0.1 V is applied between the gold wire and the metallic layer deposited on the backside of the sample (through the printed circuit board provided with electrical contact).

When reaching the desired pore diameter, the etching process is stopped, the solution is removed from the cell and the chamber is rinsed with distilled water. To completely remove the traces of sodium hydroxide from the cell, a dilute aqueous solution consisting of 10 ml 37% HCl per litre is left in the cell for 30 minutes. Subsequently, the cell is washed again and then filled with the solution for electrodeposition. We used a Watts solution with 225 g/l $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, 30 g/l $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ and 30 g/l H_3BO_3 .

The electrochemical deposition of the Ni wires is performed in a two-electrode arrangement with a nickel anode, the gold layer on the backside of the foil acting as a cathode. The wire is grown at a potential difference of 1 V between anode and cathode. When the wire reaches the membrane surface, a cap starts to grow on top. At this point in time, the electrodeposition process is stopped, the sample removed from the cell and washed with distilled water. To contact the wire, a thin gold layer is sputtered on the top surface of the polymer. In this way, a good and stable contact is established, allowing electrical measurements to be performed on the wires. The wire resistance was determined via I-V characteristics. We also measured the resistance as a function of an external magnetic field.

Embedding the samples in epoxy resin increased the lifetime of the nanowires to several months of storage without changing their properties. The same etching and growth procedure was also applied to foils irradiated with 10^6 ions/cm². The polymer templates were subsequently dissolved in CH_2Cl_2 , and the shapes and diameters of the wires were determined by means of scanning electron microscopy (SEM).

3. Results and discussion

The SEM micrographs of wires grown in multi-porous membranes showed that these wires are conical, the opening angle depending on the etching conditions. Composition, concentration and temperature of the etching solution determine the size and geometry of the resulting pores, the pore diameter increasing linearly with etching time. Fig. 2 illustrates this aspect for nickel nanowires grown in polymer pores that were produced at the same temperature (50°C), but each with different etching times.

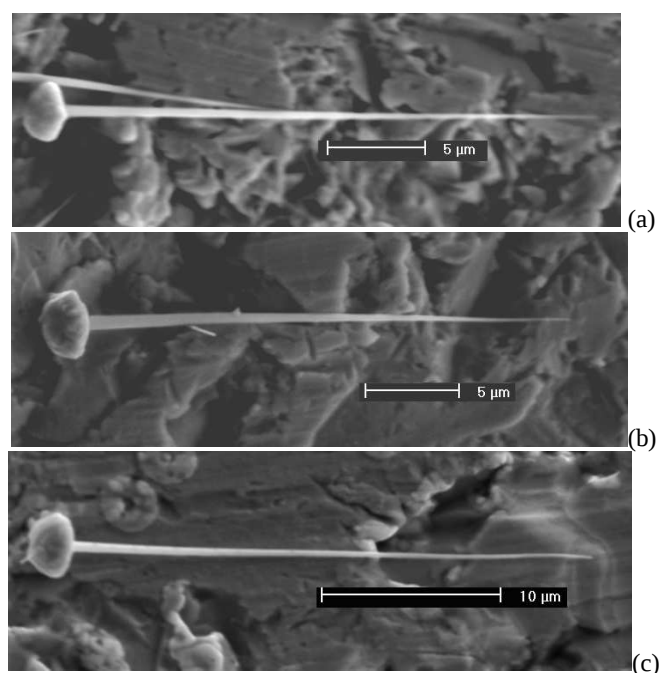


Fig. 2. SEM images of conical wires grown in porous membranes etched in solutions of 5M NaOH and 50 vol.% methanol. The etching times and approximate diameters are: (a) 400 s, 80 and 550 nm; (b) 450 s, 100 and 700 nm; (c) 500 s, 140 and 800 nm.

The voltage applied and the current recorded during etching are presented in Fig. 3. The average breakthrough time (the time at which the pore is completely etched through and an electrical current can be measured) was 250 ± 25 s for all pores. Fig. 3 represents the etching process of a pore corresponding to the wires displayed in Fig. 2.

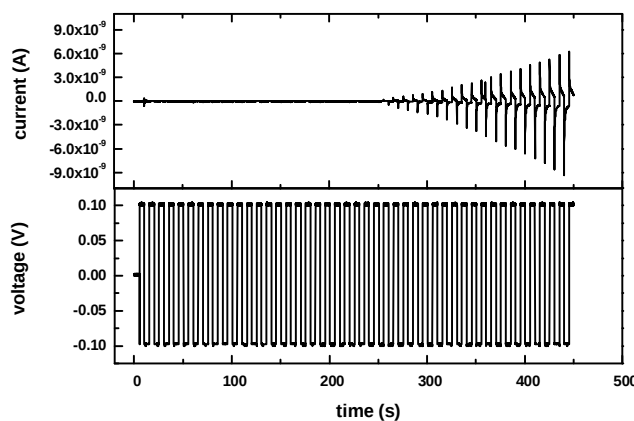


Fig. 3. The voltage applied and the current recorded during etching of a single pore with 5M NaOH and 50 vol. % methanol at 50 °C for 450 s.

The typical behaviour of the current recorded as a function of time during single-wire growth is presented in Fig. 4. As indicated in this figure, the wire growth comprises three steps: formation of an electrical double layer, growth of the wire inside the pore, and growth of a cap after the wire reaches the top of the pore. The sharp current increase by several orders of magnitude after about 200 s corresponds to the strong increase of the surface participating in the process, associated with the completion of pore filling and the beginning of cap growth.

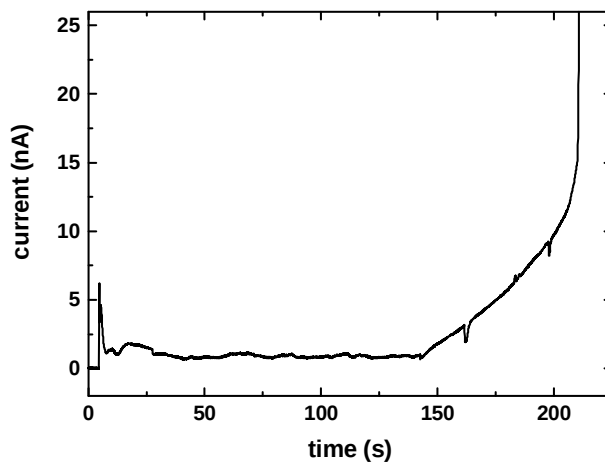


Fig. 4. Current recorded during wire growth in a pore resulting from 450 s etching with 5M NaOH and 50 vol. % methanol. The voltage was 1 V.

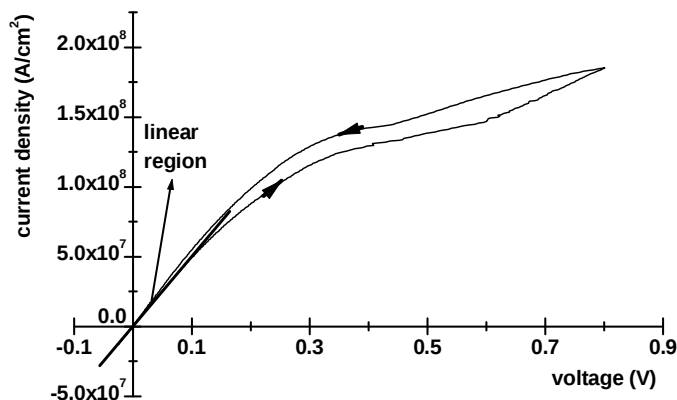


Fig. 5. The current-voltage characteristic of a Ni nanowire acquired in 50 μA steps of length 10 s, with linear behaviour up to 10^8 A/cm^2 .

The current-voltage characteristic of a Ni nanowire (see Fig. 5) shows a linear behaviour for current densities smaller than 10^8 A/cm^2 . When increasing the current rapidly, a hysteresis occurred for large current densities, presumably caused by heating of the nanowire. The maximum current density a single Ni wire could carry was found to be $3 \times 10^8 \text{ A/cm}^2$.

Magnetoresistance measurements were also used to investigate the properties of single Ni wires. We observed no variation of resistance when an external magnetic field was applied parallel to the wires. For a field applied perpendicular to the wire axis, the maximum resistance change was about 1%. Fig. 6 shows one of the first results obtained for a Ni wire of 80 nm in diameter at the tip, the electrical current being applied along the axis of the wire.

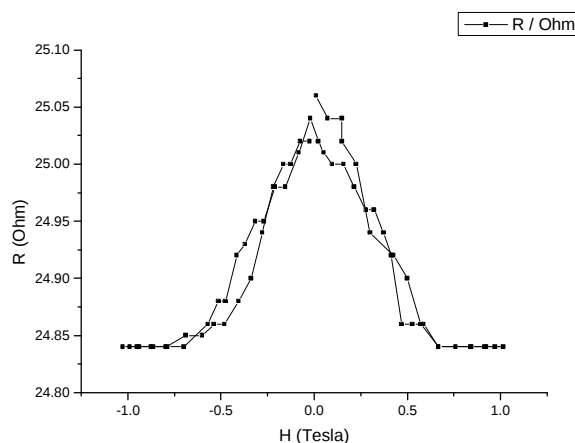


Fig. 6. The electrical resistance of a single Ni nanowire versus a magnetic field applied perpendicular to the wire axis.

4. Conclusions

Single-pore membranes were prepared by irradiating polymer foils with exactly one swift heavy ion. By one-side etching, conical pores of different diameters were created in these membranes which acted as templates for the growth of single Ni nanowires.

The nanowires were provided with electrical contacts by a simple and straight-forward method. The electrical measurements indicated that the wires have a low contact resistance and are

of an excellent quality, as they can sustain considerably high current densities. First magnetic measurements proved the occurrence of anisotropic magnetoresistance.

Our new method of contacting single nanowires offers the possibility of measuring transport properties of a wire without the use of lithographic processes or further manipulation while a wider range of growth conditions for the nanowires becomes accessible.

Acknowledgements

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References

- [1] C. R. Martin, *Science* **266**, 1961 (1994).
- [2] A. Fert, L. Piraux, *J. Magn. Magn. Mat.* **200**, 338 (1999).
- [3] M. E. Toimil-Molares, V. Buschmann, D. Dobrev, R. Neumann, R. Scholz, I. U. Schuchert, J. Vetter, *Adv. Mater.* **13**, 62 (2001).
- [4] P. Apel, R. Spohr, Introduction to ion track etching, www.iontracktechnology.de.
- [5] I. Enculescu, Z. Siwy, D. Dobrev, C. Trautmann, M. E. Toimil Molares, R. Neumann, K. Hjort, L. Westerberg, R. Spohr, *Appl. Phys. A* **77**, 751 (2003).
- [6] L. Piraux, S. Dubois, J. L. Duvail, K. Ounadjela, A. Fert, *J. Magn. Magn. Mat.* **175**, 127 (1997).
- [7] L. Piraux, S. Dubois, E. Ferain, R. Legras, K. Ounadjela, J. M. George, J. L. Maurice, A. Fert, *J. Magn. Magn. Mat.* **165**, 352 (1997).
- [8] R. Ferre, K. Ounadjela, J. M. George, L. Piraux, S. Dubois, *Phys. Rev. B* **56**, 14066 (1997).
- [9] J.-E. Wegrowe, D. Kelly, X. Hoffer, Ph. Guittienne, J.-Ph. Ansermet, *J. Appl. Phys.* **89**, 7127 (2001).
- [10] A. Bachtold, C. Terrier, M. Kruger, M. Henry, T. Hoss, C. Strunk, R. Huber, H. Birk, U. Staufer, C. Schonenberger, *Microelectron. Eng.* **41/42**, 571 (1998).
- [11] L. Gravier, J.-E. Wegrowe, T. Wade, A. Fabian, J.-Ph. Ansermet, *IEEE Trans. Mag.* **38**, 2700 (2002).
- [12] M. E. Toimil Molares, E. M. Höhberger, Ch. Schaefflein, R. H. Blick, R. Neumann, C. Trautmann, *Appl. Phys. Lett.* **82**, 2139 (2003).
- [13] Y. Huang, X. Duan, Q. Wie, Ch. Lieber, *Science* **291**, 630 (2001).