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# Magnetic nanoconstrictions made from nickel electrodeposition in polymeric bi-conical tracks: Magneto-transport behavior

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## HIGHLIGHTS

- Single nanoconstriction per magnetic wire is obtained by bi-conical track etching and electrodeposition.
- Magnetoresistance measurements at various angles result in irreversible jumps.
- Resulting jumps of magnetization have been attributed to the pinning and depinning of a constrained magnetic domain wall.

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## ABSTRACT

In a cylindrical magnetic nanowire, a magnetic domain wall (DW) can move along the wire when an applied magnetic field or a spin-polarized current is applied. We show that in a magnetic device composed of two conical nanowires connected by a nanosized constriction, a DW can be trapped and detrapped. The magnetoresistance and the relaxation processes of the DW exhibit a specific behavior. Such a device has been fabricated by Ni electrodeposition in bi-conical tracks polymer membrane made of Swift Heavy Ions bombarded poly(VDF-co-TrFE) copolymer and poly(ethylene terephthalate) PET thin films. The latter method allows to monitor the conicity of the bi-conical wires and to give access to a panel of very well-defined structures.

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## 1. Introduction

In the frame of the recent development of a spin-based electronics, domain walls (DWs) in magnetic nanostructures have been at the heart of a large research activity especially since the 1990s due to their potential applications in logic and memory devices (Tatara et al., 1999; Biziere et al., 2013).

P. Bruno (Bruno, 1999) has investigated theoretically the properties of a geometrically constrained magnetic wall in a constriction separating two wider regions. He has shown that the structure and the properties of such a wall differ considerably from their unconstrained counterparts. In particular, the wall width of a geometrically constrained magnetic wall can become

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very small if the characteristic length of the constriction is small, as is actually the case in a nanoconstriction. Many researchers have studied the effect on magnetoresistance MR in atomic point contacts (Gabureac et al., 2004; Viret et al., 2002; Montero et al., 2004). Depend on the studied nano-object, the geometry alone does not completely satisfy the explanation of MR experimental results and magnetoelastic effects should not be neglected (Wegrowe et al., 2003).

Such an attractive physical phenomenon still motivates today the researchers in the field. Atomic point contacts are so difficult to obtain that they are not yet viable for industrial applications. Similar effects have been reported on nanosized contacts or constrictions in spin-valves (Noh et al., 2011). Different techniques have been used to fabricate nanosized constrictions: most of them are created by e-beam lithography with various methods by placing notches on a magnetic wire (Zambano and Pratt, 2004). Other patterns are obtained by first designing wide Ni tracks by UV lithography in which are defined nanoconstrictions of

20–40 nm wide by several techniques including electrodeposition (Cespedes et al., 2002), electron beam lithography, and focused ion beam FIB milling (Cespedes et al., 2005). Silica templates are also used (Surowiec et al., 2007).

Here, we investigate a novel way to obtain nanoconstrictions suitable for such physical measurements: the track-etch technique in polymers. Generally, track-etched polymer films are performed in conditions leading to cylindrical track shapes. Playing on the ratio ( $v_t/v_b$ ) of track and bulk etch-rates, bi-conical tracks can be easily obtained. The electrodeposition technique is then used to fill the bi-conical tracks. We have recently used this to realize bi-conical Ni nanostructures from poly(ethylene terephthalate) PET matrix (Biziere et al., 2011). However, it was found difficult to decrease the constriction size below several tens of nanometers because the PET sensitivity to hydrolysis in the track core was too high, resulting in a non-constant value for  $v_b$ . In the present work, we have found a polymer matrix that exhibits a constant  $v_b$  value; thus, the resulting controlled  $v_t/v_b$  ratio allows us to create bi-conical track shapes with well-defined nanosized constrictions a few nanometers in diameter.

These bi-conical Ni nanostructures were further contacted and we also report herein a qualitative description for some remarkable magnetic field induced changes of their resistance.

## 2. Materials and methods

### 2.1. Materials

10  $\mu\text{m}$  thick Poly(VDF-co-TrFE) thin films were purchased from PiezoTech SA company. 16  $\mu\text{m}$  thick poly(ethylene terephthalate) thin films of from GoodFellow were furnished by Emmanuel Balanzat (CIMAP at GANIL, Caen, France). Potassium hydroxide, potassium permanganate, potassium disulfite, acrylic acid (AA), Mohr's salt ( $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ ) and sodium hydroxide were purchased from Sigma-Aldrich. Alexa Fluor<sup>®</sup> 488 hydrazide ( $\text{C}_{21}\text{H}_{15}\text{N}_4\text{NaO}_{10}\text{S}_2$ ) was purchased from Invitrogen.

### 2.2. Irradiation

Polymer films were used without pre-treatment. Swift heavy ion (SHI) irradiations were performed at GANIL, Caen. Films were irradiated with  $\text{Kr}^{36+}$  ions (10.37 MeV/amu, fluence from  $10^6$  to  $10^7 \text{ cm}^{-2}$ ) under He atmosphere. Samples were stored at  $-20^\circ\text{C}$  under  $\text{N}_2$  atmosphere until chemical etching.

### 2.3. Chemical etching

SHI-irradiated poly(VDF-co-TrFE) films were chemically etched using permanganate solution (0.25 M) in a highly alkaline aqueous solution (KOH, 10 M) at  $80^\circ\text{C}$  with different etching times from 0.5 to 2.5 h. Obtained membranes were washed in potassium disulfite solution (15%), then, washed 3 times with deionised water and dried at  $50^\circ\text{C}$  under vacuum. SHI-irradiated PET films were chemically etched using sodium hydroxide solution (NaOH, 2 M) at  $80^\circ\text{C}$  at various etching times from 5 to 20 min. Obtained membranes were washed 5 times in deionised water and dried at  $50^\circ\text{C}$  under vacuum.

### 2.4. Electrodeposition

A piece of track-etched membrane for which one side has been sputtered with a 150 nm gold layer and the other side with 10 nm gold layer is immersed into an aqueous electrolytic solution composed of  $\text{NiSO}_4$  and  $\text{H}_3\text{BO}_4$ . The electric potential between the thicker golden surface of the track-etched membrane (working

electrode) and an Ag/AgCl reference electrode is set to  $-1 \text{ V}$ . Electrodeposition time is set at the current intensity increase when a Ni wire contacts both golden sides of the membrane.

## 3. Synthesis of polymeric bi-conical tracks

### 3.1. Angular deviation of ion tracks

Commercially available PC or PET membranes made by track-etching usually exhibit some non-negligible angular deviations due to the rolling system when irradiating on large scale. The experimental set up at GANIL allows irradiating polymer films on flat holders normal to the beam direction limiting the angular deviation. Track-etched membranes are obtained by Swift Heavy Ions (SHI) irradiation of polymer films and post-irradiation chemical etching of the ions tracks (Chmielewski et al., 2007). This well-known technique leads to monodisperse cylindrical shaped pores. The SHI irradiation was performed at GANIL using the SME beam line. The energy range used in the present paper was 10 MeV/amu to cross homogeneously all the fluorinated polymer films thickness (around 10  $\mu\text{m}$ ). Under such conditions all ion tracks constitute a continuous trail of excitation.

Parallelism of pores has been taken into account in our experiment by irradiated polymers bands scotched on flat holders normal to the beam direction. The parallelism is then directly related to ion track divergence that is mainly due to the placement of a steel window in front of the sample during irradiation. This divergence has been evaluated by confocal laser scanning microscopy (CLSM) imaging of a membrane impregnated by a solution of fluorescent molecules (Alexafluor). This technique provides three dimension images of structures or objects labeled by fluorescent molecules beforehand. Laser causes fluorophores excitation and the consecutive fluorescence of the focal volume (voxel) is measured. Three dimensional scanning of this voxel provides 3D images. Fig. 1 shows a typical slice obtained in the  $xy$ -plane (top of figure) and the projection of all slices in  $xz$ -plane (bottom). Note that the pores radius is smaller than the microscope resolution but fluorescence emission permits to locate them. Clearly, the pores are not rigorously perpendicular to the membrane surface and are also not parallel to one another. For each slice, the positions of fluorescence spots in the  $xy$ -plane have been measured as a function of  $z$ , allowing us to compute the distribution of pore angles with respect to their mean orientation (Fig. 1). A Gaussian best fit of this distribution leads to:  $\sigma_\varphi = 0.023 \text{ rd}$ .

This result shows the very small angular deviation of the used set up.

### 3.2. Bi-conical tracks revealing

To reveal tracks by chemical etching, the track etch-rate  $v_t$  should be at least 10 times higher than the bulk etch-rate  $v_b$ . In order to tune the geometry, the best is to play both on the polymer chemistry composition to monitor its behavior towards hydrolysis ( $v_b$ ) and on the track damages by sensitization techniques such as UV lightening ( $v_t$ ).

If the polymer sensitivity towards hydrolysis is high, then the bulk etch-rate becomes closer to the track etch rate and bi-conical structures are favoured (Scheme 1). Polyesters (polycarbonates PC and poly(ethylene terephthalate) PET) are naturally good candidates. Additionally, we have also found interesting alkyl fluorinated copolymers, notably poly(VHF-co-TrFE), which can undergo double cone track etching by combining the action of a strong oxidant to create hydrolysable bridges and strong alkaline etchant to cut the bridges inducing polymer chain scissions.

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