Physical properties of magnetic metallic nanowires and conjugated polymer nanowires and nanotubes

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Abstract: The unique physical properties of nanowires and nanotubes originate from either the small dimensions of the objects, or structural modifications induced by the confined synthesis and surface effects. These features make them very promising for applications in nanoelectronics and nanosensors in particular. In this paper, we review our results on magnetic metallic nanowires and conjugated polymer nanowires and nanotubes prepared by the template method. Their magnetic, electrical and optical properties differ from the bulk counterparts. Mechanisms responsible for these differences have been identified.

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Keywords: nanowire; nanotube; nanomagnetism; conducting polymer.

Reference to this paper should be made as follows: Duvail, J-L., Dubois, S., Demoustier-Champagne, S., Long, Y. and Piraux, L. (2008) 'Physical properties of magnetic metallic nanowires and conjugated polymer nanowires and nanotubes', *Int. J. Nanotechnol.*, Vol. 5, Nos. 6/7/8, pp.838–850.

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1 Introduction

The widespread interest in nanomaterials originates from the fact that their properties are usually different from those of the bulk materials. These phenomena arise from quantum confinement, finite size effects as well as nano-sized filler effect. The ability to selectively tune defects, electronic states and surface chemistry has motivated the

development of various methods to fabricate metallic, inorganic and polymeric nanomaterials. For magnetic nanowires, new phenomena are due to dimensions comparable or smaller to scaling lengths in magnetism and spin polarised transport. Interesting and novel properties include original transport and magnetic properties such as giant magnetoresistance [1], magnetisation reversal in a single nanowire [2,3], domain wall magnetoresistance [4], quantised spin transport in nanoconstrictions [5], tunable magnetic anisotropy [6], resonance frequency tunability with potential technological applications in microwave devices [7]. For conjugated polymer-based nanowires and nanotubes, nanoscale size control leads to superior physical and chemical properties in relation to the molecular and supermolecular structures [8,9]. The wide range of source materials, electrical and optical behaviour and tunable surface functionalities make these systems very promising for chemical and bio-sensors, organic electronics, supercapacitor, photovoltaic cells, electrochromic devices [10].

Here, we review our results on magnetic metallic nanowires and conjugated polymer nanowires and nanotubes prepared by the template method. Their magnetic, electrical and optical properties differ from the bulk counterparts. Mechanisms responsible for these differences have been clearly identified.

2 Synthesis: the template method

The hard-template method, which consists in filling the void spaces of a host porous medium with one or several desired materials using a number of techniques, is now routinely used to fabricate one-dimensional nanowires and nanotubes. There are many materials such as zeolithes, nanoporous glass, block copolymer films which have been and are currently being used as templates for the fabrication of nanomaterials, but particle track-etched membranes (PTM) and anodic aluminum oxide (AAO) templates are the most commonly used nanoporous materials. The 'track-etching' method consists in bombarding a polymer (polycarbonate, polyester, polyimide, ...) film (typically 5-50 µm thick) with heavy energetic ions in a cyclotron to create damage tracks in the material. The bombarded film is then irradiated with UV light for increasing the chemical etching selectivity. Tracks are finally etched by a basic aqueous solution into randomly distributed monodisperse pores (Figure 1(a)). Though these membranes are commercially available, the technology has been improved at the POLY lab at the UCL in order to produce reliable and reproducible nanoporous polymer membranes with improved properties in terms of pore shape, minimal pore size, pore size distribution and arrangement of pores across the membranes [11]. The technology has now recently been transferred to it4ip (spin out from UCL) [12] that produces membranes with mean pore size diameter ϕ in the range 10 nm to a few μ m, low pore size distribution, perfectly cylindrical pores with parallel arrangement across the membrane. With such optimised properties, nanoporous PTM are excellent templates for the preparation of arrays of nanostructures with uniform dimensions. The track-etching process has also been adapted for the fabrication of supported nanoporous templates made of a thin spin-coated polymer layer (thickness from 200 nm to several microns) on a metal-coated substrate (glass slide, silicon wafer, etc.) [13]. Supported templates with pore size ranging from 10 nm to 100 nm are now available and successfully used for the synthesis of vertically aligned free standing nanowires and nanotubes and are promising systems for the fabrication of various devices [14].



Figure 1 SEM pictures of: (a) PTM template surface; (b) AAO template surface; (c) array of conjugated polymer nanotubes and (d) free metallic nanowires

Porous alumina films formed by anodic oxidation of aluminum in an acidic solution have also been intensively used to fabricate nanowires. Pore densities as large as 10^{12} cm⁻² and pore diameters ranging from approximately 5 nm up to >300 nm can be produced. AAO membranes are commercially available in a limited number of pore diameters. It has also been shown that a two step anodisation process can lead to highly ordered two dimensional pores arrays with a hexagonal pattern (Figure 1(b)) [15]. The fabrication of alumina templates supported on Si substrate, showing improved thermal and mechanical stability, have been recently developed at the PCPM lab at UCL and IMN.

Several chemical and electrochemical strategies allowing the deposition of a material within the pores of a template have been encountered in the literature. Each strategy has to fill some criteria:

- the precursor solution has to wet the nanopores
- the template should be chemically and thermally stable under the material synthesis conditions
- the deposition rate should not be too fast to avoid pore blockage at the membrane surface before formation of nanowires or nanotubes.

Electrochemical deposition offers marked advantages over other methods for the synthesis of 1D nanostructures as it does not require expensive instrumentation, high temperature or low-vacuum pressures and is not limited to a small area. Moreover, it allows the synthesis in high yield of nanotubes (Figure 1(c)) or nanowires (Figure 1(d)) with accurately controlled length by adjusting the electrodeposition time. Many different single metals (e.g., Au, Ag, Pt, Pd, Sn, Cu, Ni, Co, Pb, Bi), alloys (e.g., NiFe, CoFe, CoPt) and multilayers (e.g., Co/Cu, NiFe/Cu) and conjugated polymers (e.g., polypyrrole PPy, polyaniline PANi, polythiophenes) have already been electrosynthesised in hard templates. Regardless of the type of material to be deposited, the first step of the

electrochemical process consists in coating one face of the template with a sufficiently thick evaporated metal film to cover the pores. This metal film serves as working electrode. A negative potential (reduction) is applied to the system for metal deposition while a positive potential (oxidation) is applied for polymer electrosynthesis. After deposition of the desired components, the nanostructures can be kept inside the pores or they can be freed from the template membrane and collected as an ensemble of free nanoparticles (Figure 1(d)). Alternatively, if nanostructures are synthesised within a supported porous thin film and the template is removed, an ensemble of nanostructures that protrude from the surface like the bristles of a brush can be obtained (Figure 1(c)).

3 Magnetic properties of magnetic metallic nanowires

In this section, we have chosen to focus on magnetisation reversal of single nanowires. The direct study of individual nanowires magnetic properties requires very sensitive techniques such as micro-superconducting quantum interference devices (SQUID's) [2]. However, a limitation of this technique is the low temperature range in which measurements must be performed. Resistance measurements performed as a function of the applied field may advantageously be used, at room temperature, to probe the alignment of the magnetic moments on the length scale of the electronic mean free path. Indeed, it is well known that magnetic materials resistance depends on the relative orientation of the current and magnetisation [16], the so-called anisotropic magnetoresistance (AMR). AMR is due to the anisotropy of spin-orbit scattering in transition ferromagnetic metals and leads to changes in the resistivity as the angle between the current lines and magnetisation is modified. The total change in resistivity between the high resistance state where the magnetisation is parallel to the current and the low resistance state where the magnetisation is perpendicular is usually small (a few percents or even less). Nevertheless, as resistance measurements can be performed with high accuracy, AMR was used to study the magnetisation reversal of magnetic nanowires such as Ni and Co at various temperatures [3,17] and the results were quantitatively analysed using classical magnetisation reversal theories. The resistance variation with magnetic field is related to the magnetisation M(H) by:

$$R(H) = R_0 + (\Delta R)_{\max} \left(\frac{M(H)}{M_s} \right)^2$$
(1)

where $M(H) = M_s \cos[\theta(H)]$ is the magnetisation measured along the wire axis, M_s is the saturation magnetisation and θ is the angle between the applied field and the current (which is along the wire axis). In equation (1) $(\Delta R)_{max}/R_0$ defines the AMR ratio.

Figure 2 shows the magnetoresistive hysteresis loops recorded at room temperature on a single 75 nm Ni nanowire for the angles $\theta = 10^{\circ}$ and $\theta = 75^{\circ}$. These AMR data were obtained on a single 20 µm long Ni wire embedded in the polymer membrane, using the self-contacting method described in [3]. Low density arrays of Ni nanowires constitute a simple magnetic system in the sense that both magnetocrystalline anisotropy and dipolar interactions can be neglected [18], so that only shape anisotropy should be taken into account. Taken as a whole, the AMR curves are composed of continuous resistance variations, which correspond to reversible rotation of the magnetisation and of discontinuous jumps of resistance at characteristics fields, which correspond, to magnetisation reversal. The reversal process starts with nucleation of a reversed domain at the end of the wire [19]; the reversed domain thus propagates to the other end. Resistance jumps, observed in AMR curves, refer to successive pinning and depinning of the domain wall. Inset of Figure 2 shows a zoom in the region where the discontinuity occurs. Two jumps of resistance are evidenced at two fields close to each other. The first jump of resistance refers to the nucleation of the magnetisation reversal and to the propagation of the domain wall until it is trapped at a pinning centre. The domain wall is evacuated only at a larger field (giving rise to a second jump) where the magnetisation is completely reversed. Such a behaviour has been observed in most investigated samples. Magnetisation reversal achieved in several steps probably results from structural defects acting as pinning centres. From the angular dependence of the nucleation field, it has been shown that reversal mechanism in Ni nanowires is consistent with a curling rotational mode [19]. Moreover, the magnetisation reversal is initiated in a volume much smaller than that of the whole wire despite the high aspect ratio of the studied wire which can be considered as infinite cylinder [3,17].

Figure 2 AMR characteristics at 300 K of a single 75 nm Ni nanowire for two angles between the wire axis and magnetic field direction. Inset: zoom showing the irreversible jumps of the resistance as the magnetisation is reversed (from [3])



At low temperature, nucleation field values give evidence, in the case of Ni nanowires, for an extra uniaxial anisotropy adding to shape anisotropy. Such a result has been confirmed by hysteresis loops measurements. Indeed, the extra-uniaxial anisotropic energy, represented in Figure 3 as a function of the pertinent parameter of the problem (i.e.: the radius ratio $k = R_{Ni}/R_{PC}$), R_{Ni} being the radius of a single Ni wire and R_{PC} the equivalent radius of polycarbonate cylinder), has been estimated from the change in magnetisation work calculated for applied field parallel and perpendicular to the wire axis. It has been shown, from XRD analyses, that thermal stresses and induced magnetostrictive effects can give account of the extra uniaxial anisotropy encountered at low temperature. Calculation of the thermal stress and deformation components have shown that magnetostrictive effect (dotted line in Figure 3(left)) are conjointly induced by the PC and Ni thermal expansion coefficients mismatch and by the low volume fraction of Ni in our Ni/PC composites.





Cobalt nanowires have focused much attention as a consequence of the large crystal anisotropy developed in the Co-*hcp* structure. Under usual electrodeposition conditions, Co nanowires exhibit a polycrystalline structure with a hexagonal close packed (*hcp*) structure for most grains. The hexagonal axis of these grains, whose size can be larger than one micron, lies within a few degrees normal to the wire axis and are randomly distributed. In this case, the effective anisotropy decreases as the large and temperature-dependent magnetocrystalline anisotropy competes directly with the shape anisotropy. Furthermore, the coexistence of varying amounts of the two cobalt phases (hcp and fcc, the latter showing no significant magnetocrystalline anisotropy) was found by several groups. Figure 3(right) shows the magnetoresistance recorded at room temperature in a 0.5 µm long segment of an isolated 60 nm Co nanowire for both parallel and perpendicular direction of the applied magnetic field with respect to the wire axis [20]. A completely different behaviour than those observed on Ni nanowires (Figure 2) is observed. Indeed, for the Co sample, magnetoresistance is much larger in the perpendicular configuration. After either perpendicular or parallel saturation, the same low resistance state at zero field is obtained, with a value close to the one obtained for perpendicular saturation. These observations suggest that a longitudinal single domain state cannot be achieved in zero field although the very elongated shape of the objects favours such a magnetic configuration. However, a complex multidomain pattern, in which the domain magnetisation has a strong transverse component, is always formed in zero field. Such a complex multidomain configuration is favoured by the perpendicular to the wire axis magnetocrystalline anisotropy $(5 \times 10^6 \text{ erg/cm}^3)$ which competes with the parallel to the wire axis shape anisotropy $(6 \times 10^6 \text{ erg/cm}^3)$. Such a multidomain configuration has moreover been confirmed by MFM observations [21] and micromagnetic calculations [19]. For thin Co wires (wire diameters in the range 35–50 nm), no change in resistance is observed with the applied magnetic field lying parallel to the wire axis. Such a behaviour is comparable with the one observed over the whole diameter range for an array of Ni wires. Thus, only two magnetisation states, parallel and antiparallel to the wire axis are realised in Ni as well as in Co if the magnetocrystalline anisotropy is small or if the c-axis is oriented parallel to the wire axis, thus reinforcing the shape anisotropy.

4 Electrical properties of conjugated polymer nanowires and nanotubes

Pioneering studies on conjugated polymer nanowires prepared by the template method suggested that higher chain ordering is responsible for the improvements in the electrical properties when the diameter decreases below 60 nm [8,9]. Such a diameter effect on the electrical properties is of crucial importance from both fundamental and application points of view. This justifies recent studies on similar systems also prepared by the template method that result in a controversy, because opposite electrical behaviours have been measured [22,23]. The structural improvement for the smaller diameters has no concern with the controversy, as similar effects on the structure have also been shown. But it should result from two points: the real diameter of the nanowires and the electrical configuration for the measurements. Concerning the nanofibre diameters imposed by those of the pores, it is currently admitted that the pores of the commercial polycarbonate templates involved in most studies have a cigar-like shape and a rough surface. As a result, the nominal diameter given by the provider is only correct at both extremities. But for the smallest diameters, the real nanofibre diameter is two or three times larger [24] and thus it may affect some conclusions. Concerning the electrical configuration, it has to be mentioned that, up to now, all the studies addressing the diameter effect used a two-probe configuration on nanofibre arrays or four-probe geometry on a pellet, not a single nanofibre. Thus, the conductivity values can only be estimated from an approximate number of nanofibres measured simultaneously. Moreover, the contact resistance may play a role. These two points can result in significant uncertainties on the conductivity values.

In our studies, the diameter values really correspond to the diameter along the whole nanofibre. Indeed, nanoporous polycarbonate membranes with improved features were used as templates (see part 2). Nanowires of poly(3,4-ethylenedioxythiophene) (PEDOT) and nanotubes of polyaniline (PANi) and polypyrrole (PPy) have been prepared by electrochemical synthesis in polycarbonate templates with diameters ϕ ranging from 25 nm to 200 nm. Our optical and spectroscopic studies reviewed below confirm the molecular and supermolecular improvement when the diameter decreases, as observed elsewhere [8,9]. We showed that the electrical behaviour strongly differs for nanowires still embedded in the membrane and for individual nanowires measured after removal of the template. Changes in the doping level induced by the confined synthesis can account for this difference.

4.1 Spectroscopic studies

Spectroscopies methods are powerful tools for elucidating the molecular and electronic structure of conjugated polymers. Our studies on PANi, PPy and PEDOT nanowires and nanotubes by Raman, absorbance and electron spin resonance spectroscopy arrive at the same conclusions: the smaller the nanofibre diameter, the larger the chain ordering and the conjugation length. As an example, typical Raman spectra of polypyrrole (PPy/ClO₄) film and nanotubes of different diameters are shown in Figure 4(left). The ratio between the intensity of a band sensitive to the oxidation state of the polymer (1595 cm⁻¹) and the intensity of the skeletal band (1500 cm⁻¹) was used to obtain a qualitative measurement of the relative conjugation length. Taking into account the experimental errors due to the poor resolution of the 1500 cm⁻¹ band, a continuous increase of the 1595/1500 intensity ratio was observed when going from PPy films to the narrowest PPy nanotubes

(ϕ = 35 nm). The ring deformation mode and the symmetric CH-in plane bending modes are splitted due to contributions from the radical cations (polarons) and dications (bipolarons): peaks at 934 cm⁻¹ and 1086 cm⁻¹ are associated with the bipolaron structure and those at 968 cm⁻¹ and 1055 cm⁻¹ are associated to the polaron structure. Different bipolaron/polaron band ratios were calculated and the higher dication segments content was always found for the narrowest PPy nanotubes [25].





For understanding the electrical properties, it is crucial to estimate the doping level of the nanowires and nanotubes. XPS studies on as-grown PEDOT nanowires and PPy nanotubes indicate that the doping level is reduced by a factor of 2-4 for diameters smaller than typically 50 nm in comparison to the film case [23,25]. This effect could be linked to the limited diffusion of species for the template electropolymerisation. Moreover, UV-Vis spectroelectrochemistry studies on PPy, PANi and PEDOT nanowires and nanotubes after the template removal [25,26] showed an enhanced electroactivity for the smaller diameters. When the diameter is smaller, a higher (resp. lower) contribution of bipolarons has been evidenced in the doped (resp. neutral) state at a fixed potential of $E_a = +0.80 \text{ V/Ag/AgCl}$ (resp. $E_a = -0.80 \text{ V/Ag/AgCl}$). This enhanced electroactivity is also confirmed by the comparison of the absorption spectra in the reduced state ($E_a = -0.80 \text{ V/Ag/AgCl}$) for the PEDOT film and the PEDOT 30 nm diameter nanowires (Figure 4(right)). The residual absorption due to polaron structure that appears in the range 800–1000 nm for the film, is strongly reduced for the 30 nm nanowires. This study indicates that, after the membrane removal, both the fully neutral and doped states can be reached due to the easier counter-ion diffusion as the nanowire diameter is smaller. These results are of primary importance to analyse the electrical measurements.

4.2 Electrical properties

Arrays of PPy nanotubes and PEDOT nanowires were measured in a two-probe geometry (Figure 5(left)). Surprisingly, it was found that a strong increase in the resistivity ratio

(PEDOT nanowires: R(1.5 K)/R(300 K), PPy nanotubes: R(77 K)/R(300 K)) when the diameter decreases, which indicates an insulating behaviour more pronounced [23]. This result, in apparent contradiction with the improved structure evidenced above, contrasts with previous studies [27] but corroborates the results of Mativetsky and Datars [22]. Recently, we determined the electrical behaviour of isolated PEDOT nanowires connected by four platinum leads deposited by focus ion beam [28]. Both the strong increase of $\sigma(300 \text{ K})$ and the fall of $\rho(10 \text{ K})/\rho(300 \text{ K})$ by five orders of magnitude indicate that the smaller diameter nanowires are much more conductive (Figure 5(right)). Moreover, a precise analysis gives evidence for an insulating regime as the diameter is large (190 nm), a critical regime for intermediate diameter (100 nm) and a metallic regime for a diameter equals to 35 nm [28]. This tendency is consistent with the improved molecular and supermolecular structure for the smaller diameters, as discussed above. Different doping levels for nanowires still embedded in the polycarbonate template on the one hand, for isolated nanowires on the other hand, could explain the opposite electrical behaviours, as discussed below.





A first point to consider is the effect of the solvent (dichloromethane) used to dissolve the PC membrane. We first checked by a XPS study on PEDOT films that the doping level is not modified by the solvent. Concerning PEDOT nanowires, XPS studies have shown a strong decrease in the doping level when the diameter is reduced, that can explain the more insulating behaviour for the nanowires still embedded in the membrane [23]. Otherwise, the spectroelectrochemical studies demonstrated that, after the membrane removal i.e., when the whole nanowire surface is accessible, the fully doped and neutral states can be reached easily. It has also been shown that PEDOT films kept in air are stable in the doped state. We verified that the conductivity of isolated PEDOT nanowires stay unchanged during six months. As a consequence, it is reasonable to consider that, when dispersed on a wafer and kept in ambient conditions, small diameter PEDOT nanowires are fully doped. The doping by organic species resulting from the decomposition of the platinum precursor during focus ion beam deposition of the electrodes could also be involved. Then, the electrical properties of isolated PEDOT nanowires with a small enough diameter fully benefit from the improved chain ordering and the high doping level.

5 Conclusion

Magnetic metallic nanowires and conjugated polymer nanowires and nanotubes were prepared by the hard-template method. Very original physical properties have been evidenced and the mechanisms responsible for these properties have been identified: small diameters comparable to characteristic physical lengths (supra)molecular modifications induced by the confined synthesis, small size effects.

Acknowledgements

The work made at IMN was partly supported by the C'Nano Nord Ouest Network and the 'Communauté Urbaine de Nantes'. One of us (Yung Long) thanks the National Natural Science Foundation of China (Grant Nos. 10374107 and 10604038). The work made at UCL-PCPM was supported by the Interuniversity Attraction Poles Program (P6/42) – Belgian State – Belgian Science Policy. The work made at UCL-POLY was supported by the Belgian Federal Public Planning Service Science Policy (Inter-University Attraction Pole SC²), by the Belgian National Fund for Scientific Research (FNRS) and by the French Community (ARC project).

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