Cylindrical Magnetic Nanowires Applications

Julián A. Moreno¹, Cristina Bran², Manuel Vazquez², Jürgen Kosel³

¹ Physical Science and Engineering Division

King Abdullah University of Science and Technology (KAUST), Thuwal 23955-6900, Saudi Arabia

² Instituto de Ciencia de Materiales de Madrid, CSIC, Madrid, 28049, Spain

³ Computer Electrical and Mathematical Science and Engineering Division

King Abdullah University of Science and Technology (KAUST), Thuwal 23955-6900, Saudi Arabia

Cylindrical magnetic nanowires feature unique properties, which make them attractive for fundamental research as well as novel applications. These one-dimensional structures introduce a pronounced shape anisotropy that together with material selection can strongly affect the magnetic properties and can be tuned by incorporating segments of different materials or diameters along the length. They attract a large interest in the scientific community, ranging from physicists to material scientists to bioengineers. Consequently, these nanowires are developed for and employed in very diverse applications in medicine, biology, data and energy storage, catalysis or microwave electronics, among others. In this review we investigate the most active emerging applications of cylindrical nanowires grown in alumina templates by electrochemical deposition. This method has several key features, including low cost and a high level of control over the design. A fundamental property that distinguishes those applications is the operating frequency, which we chose to apply as an underlying structure for this review. With this we attempt to provide a wide and organized view of applications based on cylindrical magnetic nanowires with a focus on tailored physical and chemical properties.

Index Terms—cylindrical magnetic nanowires, nanotechnology, electroplating, sensors, devices, biomedical, passive electronic elements.

I. INTRODUCTION

CYLINDRICAL MAGNETIC nanowires (NWs) are one dimensional structures with unique fundamental properties and a wide range of novel applications. A high aspect ratio leads to large shape anisotropies, which can dominate the magnetic properties and magnetization processes. Their geometry causes a curvature-induced effective anisotropy or chiral symmetry breaking, and favors the formation of uncommon magnetic textures, contrary to more common planar nano-strips [1]–[4]. Several reviews have been published, mostly in the form of book chapters focusing on fabrication and characterization of NWs, yet there are relatively few documents with concise emphasis on the working of devices (regardless of their technological readiness). For instance, the latest works from Mohammed et al [5], Xu et al [6], Staño & Fruchart [7], L. Piraux [8] and the book edited by Vazquez [9], amongst others, are excellent references to understand the state-of-the-art of fabrication, characterization and magnetization control of NWs, setting the background of fundamental aspects. The main purpose of this review is to compile and describe the most recent applications based on NWs. Although there are several techniques to grow NWs such as using few nanometer-diameter nuclear-track etched polymer templates [8], growing them individually using Focused Electron Beam Induced Deposition (FEBID) [10] and directly released in solution using soft, polyol or organometallic chemistry [11], to name a few, we will limit our considerations to those works, in which the NWs were fabricated by electrochemical deposition into alumina templates. This last-mentioned method provides a large control over parameters in combination with many degrees of freedom for tailored designs.

These applications exploit either internal magnetization changes or external torques on individual or collective NWs with single or multi-domain states at certain frequencies:

- They can act as a static source of magnetic field [12] and therefore be detected by e.g. magnetoresistive sensors [13], [14], serve as highly sensitive magnetic force microscopy (MFM) tips [15] or induce field fluctuations in energy harvesters [16], [17].
- Hybrid magnetic / piezoelectric, magnetoelectric and magnetic nanowires are proposed for bioengineering applications as nano-robots and nano-swimmers for biomedical applications [18], [19], or for security code sensors [20].
- They can be excited with lasers or alternating magnetic fields up to hundreds of kilohertz and produce motion and/or heat [21]–[23]. This makes them attractive for biomedical applications [24], targeting cancer cells when they are properly functionalized [25], [26] or as MRI contrast agents working at around hundredths of megahertz [27], [28].
- Arrays of NWs have been used as electromagnetic pulse detectors [3], microwaves circulators or phase shifters [29]–[32] at 10 – 40 GHz frequencies.
- When isolated, effects like giant magneto-resistance (GMR) [33]–[35], domain wall (DW) pinning [36]–[38] and spin transfer torque (STT) [39]–[41] are exploited mainly for prospective memory applications [42]–[45] employing nanoseconds (GHz) pulses.

Fig 1 illustrates some of these applications at their range of operation frequency. Notice there is a gap from around

Fig 1. Overview of the wide range of applications of cylindrical magnetic nanowires and the operation frequencies.

1000 kHz (1 MHz) to the GHz regime. These are frequencies too big to induce significant mechanical movement or to be approved for use in living organisms and too small for electronics devices. Classifying applications in terms of the used frequency, either applied (external excitation) or internal (magnetization changes), as in **Fig 1**, aims to provide a structured view of the field from the fundamental research to the design/engineering perspective.

II. FABRICATION OF CYLINDRICAL NANOWIRES

One of the most common and reproducible ways of fabricating NWs is by electrochemical deposition into anodized alumina templates. These templates consist of cylindrical nano-channels through an alumina bulk with a high regularity in length and diameter. Anodization of aluminum provides a wide range of channel diameters and inter-channel distances, and some control over the geometry of the channels like diameter modulations along the length [46]–[48]. Electrodeposition of metals into these templates results in the metal filling the channels and replicating their shape. The resulting NWs can be studied inside the template or they can be partly or entirely released from it by etching the alumina. These templates also contain a high density of channels (in the order of 10^{10} channels/cm²) favoring high density arrays and fabrication yield. Therefore, these templates have been adopted for numerous applications and are widely used by research groups.

Most of the tailored properties of individual and collective NWs are inherited from the shape of the template and electrodeposition conditions. Different energy contributions define the magnetization behavior in a magnetic material, namely shape and crystal anisotropies, exchange interaction, the energy related to a self-demagnetizing field and external fields like magnetostatic interactions among neighboring nanowires. The shape of the template and length of the

nanowires defines their shape anisotropy and overall demagnetizing field, while the nature of the electrodeposited metal and the electroplating conditions define the magnetocrystalline anisotropy. This latter energy term, for example, can be tuned by a suitable choice of the pH value of the electrolyte [49] or the deposition voltage and temperature [50].

A. Fabrication of Anodized Alumina templates

The most common way of fabricating alumina templates is by anodization - controlled oxidation of pure aluminum in one of three main electrolytes: sulfuric, oxalic or phosphoric acid - driven by an electric field applied between a platinum mesh (counter electrode) and aluminum itself (working electrode) [51]. Oxidation conditions, such as temperature, electrolyte concentration and pH determine the rate at which the oxide layer is formed at the aluminum/oxide interface and dissolved into the electrolyte at the oxide/electrolyte interface. In the first anodization step (**Fig. 2a**) channels appear at random locations throughout the surface and progress towards the aluminum. These first channels appear mainly due to non-uniform oxide growth caused by the barrier oxide layer growing under tensile stress, creating cracks, where the electric field concentrates (in addition to local imperfections) facilitating oxide dissolution [48]. As this process continues, the channels increase their diameter approaching each other and depleting the aluminum sideways, eventually leading to a vertical growth in a hexagonal pattern. This depletion of the aluminum and the steady state applied electric field, define the inter-channel distance and channel diameter (**Fig. 2a - c**). These channels are also referred to as pores, even when they do not run all the way through the alumina, i.e., with both ends open.

Fig. 2 Standard anodization procedure starting with high purity aluminum with a polished surface. The first stage corresponds to the formation of random channels near the surface which then self-organize (a). This alumina from the first anodization is then removed (b), leaving a footprint as starting point for a second anodization. The second anodization results in highly ordered nanochannels with a narrow size distribution (c). The backside of the aluminum is then removed and the channels opened creating pores that run across the entire thickness (d).

To attain higher homogeneity of the distribution of the channels, the electric field that results from the voltage applied through the cell must be uniform over the anodization area. To this end, the aluminum surface is smoothed prior to anodizing to avoid electric field concentration on protuberances by electrochemically etching in a 75:25 mixture of ethanol and perchloric acid, reducing its surface roughness from the micrometer to the nanometer range [52], [53]. After this, an optional pre-patterning is performed either by removing the alumina produced during a first anodization step [54] (**Fig. 2b**) and using the resulting “footprint” over the aluminum or by indenting the surface, for instance with a stamp [55]–[59]. Stamps also allow patterns different from cylindrical shapes, such as prisms, to be produced (see [47]). Starting from the pre-patterned surface, the channels can then be grown with their length controlled precisely by the anodization time for the intended application (**Fig. 2c**). To obtain a porous template, the non-anodized aluminum at the back side and the barrier layer, formed at the end of each channel during anodization (**Fig. 2c**), are etched away (**Fig. 2d**). Variations of these stamp [60] and two-step [54] anodization procedures allow tailoring of the template’s parameters and the NW diameter along its length.

As will be seen later, for certain applications like using arrays of NWs as microwave circulators, a higher order of the NWs in the array, obtained by either of these procedures, is a key factor for obtaining sharper circulation resonant frequencies and isolation. This means, the regularity of the order has an impact on the dipolar interactions of the NWs in the array and therefore on the device performance.

By using specific anodization potentials, at least two regimes

of distinct channel diameters and inter-channel distances can be obtained for each electrolytic bath, known as mild (low-field) and hard (high-field) anodization. Whereas mild anodization potentials are directly applied and sustained, hard anodization potentials are generally ramped from the mild anodization ones. This is done to create a thick enough oxide layer that prevents the high fields from driving high local currents through the oxide layer, which would “burn” the surface [61]. Common diameters and inter-channel distance values for each bath and regime are summarized in **TABLE I** together with their respective anodization parameters. Different electrolytic baths’ characteristics such as pH, solubility or relative permittivity have an impact on pore diameter and inter-pore distance or can modify the effective electric field [48].

A quantity used to characterize the density of pores per unit area is the ratio of their (planar) surface area to the total surface area also known as porosity. In other words, it is the percentage of surface that is covered by pores. Assuming the pores are ordered in a hexagonal pattern, the porosity can be geometrically expressed as:

$$P = \frac{\pi}{2\sqrt{3}} \left(\frac{D_p}{D_{int}} \right)^2.$$

Where D_p is the pore diameter and D_{int} is the inter-pore distance, illustrated in **Fig. 2c** (A detailed calculation can be found in section 1.2.2.5 of [46]). Typical values for each bath can be found in **TABLE I**, where two trends can be recognized: porosity lowers from mild to hard anodization regimes and increases as the potential is reduced throughout the baths. This is due to the controlled oxidation rates being dependent on the strength of the electric field as discussed before.

TABLE II
THREE MAIN ANODIZATION BATHS WITH THEIR REPRESENTATIVE PORE DIAMETER AND INTER-PORE DISTANCE

ANODIZATION BATH	Regime	Potential (V)	Pore Diameter (nm)	Inter-pore distance (nm)	Porosity (%)	Reference
Sulfuric Acid	Mild	25	30	60	22.7	[65]–[67]
	Hard	70	50	130	13.4	[68], [69]
Oxalic Acid	Mild	40	35	110	9.2	[66], [70], [71]
	Hard	140	60	300	3.6	[60]
Phosphoric Acid	-	205	150	450	10.1	[65], [72]

As the electric field driving anodization is controlled by a voltage supplied from a power source, it can be varied to change the oxidation rate and, therefore, create modulations of the channel diameter [60], [62]–[64]. An example of channel diameter modulation is shown in **Fig. 3** where an electric current is applied (**Fig. 3a**), while the voltage is monitored. The process starts with mild anodization values (MA) setting the initial diameter, then, it is transitioned (TA) to hard anodization (HA) increasing the diameter to a maximum and finally returning to MA “resetting” the diameter. The resulting shape of the channel (**Fig. 3b**) follows the current profile (**Fig. 3a**) and shows that controlled diameter modulations can be achieved along the channel. This fact has been exploited to tune the optical properties of the template [63], [73] and is envisioned to be used in coatings [74] and steam generation [75], for example. For more information on applications of alumina templates, the reader is referred to the excellent reviews by Lee [48] and Sulka [47].

Fig. 3 Example of channel diameter modulation during anodization (figure adapted from [63]). This galvanostatic cycle (a) starts at mild anodization currents (MA), then is ramped up, transitioning (TA) to hard anodization (HA) and then returned to MA. The resulting channel shape is shown in (b).

It is worth mentioning that some constraints and advantages of the methods described so far. When performing indentation to pre-pattern the surface, the output area is limited by the area of the stamp or the area accessible by the milling tool in a reasonable (cost-effective) time. In addition to this, the indentations cannot deviate significantly from the self-ordered inter-pore distance obtained for each of the conditions summarized in **TABLE I**. Also, although in principle the free-standing template could be a few microns thick, it is commonly

fabricated to be around 60 μm . This is because, without a mechanical support, thinner templates are easily broken when handling them even when a frame of aluminum is kept around the anodized part. On the other hand, once anodized, the channel diameters in each template can be enlarged up to around 80% of the inter-channel distance by submerging in acidic baths [46], [76], limiting but also allowing the tailoring of porosity for specific properties. Additional steps must be performed on alumina templates to prepare them for the electrochemical growth of the NWs. One option is the deposition of a conductive thin film on one side of the porous template, shown in **Fig. 2d**, that serves as a working electrode for electroplating. In the case when such conductive film is grown in a way where it does not to fully cover the pores, the formation of nanotubes is favored (**Fig. 4b**). A different route is to start reducing the thickness of the barrier layer after the second anodization, as shown in **Fig. 2c** by progressively lowering the applied bias voltage to a small value; thus, creating “dendrites” at the end of each channel. These dendrites allow the flow of current into the aluminum for sufficiently high applied potentials, using a pulsed electrodeposition method explained below, and thus eliminate the need to remove the non-anodized aluminum as in **Fig. 2d**.

The combination of diameters and inter-channel distances, in conjunction with the possibility of widening and modulating the diameters, allows the creation of virtually any shape within those constraints. This implies that NWs grown inside the templates can also have, in principle, any shape within those constraints.

B. Electroplating of cylindrical nanowires

Electroplating of metals is used to get coatings on surfaces in several areas of industry and research [77], [78]. It consists of the reduction of metal ions from an electrolyte at a working electrode with specific conditions of temperature, pH, concentration and applied electrical bias in an electrolytic cell. Electrolytes are prepared commonly from metal hydrates in aqueous solutions, where they dissociate into an ionized metal cation and a polyatomic anion, and using chemical buffers to maintain the pH value during plating. These ions, of radii of about hundred picometers, migrate to the working electrode, due to the electric field, where they are reduced at the negatively charged surface. This allows electroplating to be performed in nano-sized electrode areas, such as those inside of the channels

of alumina templates, causing the deposited metal to acquire their shape while it grows for several tens of microns.

Most (if not all) of the electrolytes used in nanofabrication of NWs have their origin in industrial recipes and can be found in technical texts [77]. As a general rule, if an electrolyte has been used in industrial applications, it can be used for nanotechnology research. For instance, nickel electroplating is carried out with variations of the well-known “Watts” solution [79], [80], and other well-known electrolytes exist for copper [81], gold [82] and iron [83]. Nevertheless, research is carried out to improve or produce new electrolytes for nanotechnology applications [84]–[86]. For example, a single electrolyte can contain two different metallic species with different reduction potentials and can be used to deposit alternating metallic layers [33], [35], [87]. Also, intrinsic material properties are sought to be controlled while plating: crystal anisotropy can be controlled by modifying deposition pH [50], [86], [88], [89] and magnetic exchange interaction can be tailored by depositing alloys [83], [90]–[94]. One of the most desired property is single crystalline deposition, which has been achieved to a certain degree [95], [96]. These techniques are continuously being adapted and improved to obtain the needed industrial or research driven material properties.

There are mainly three methods to control the electroplated metal growth: potentiostatic, galvanostatic and pulsed electrodepositions. In the potentiostatic method, a constant voltage relative to a reference electrode is applied across the electrolytic cell. In the galvanostatic method, the current is set and the source adjusts the output voltage so as to keep that current constant with a feedback loop. In both cases, a potential high enough for the metal ions to overcome the naturally formed electrical double layer and reduce at the working electrode needs to be applied [97]. The pulsed electrodeposition method is mostly used in membranes with dendrites [98], and consist of two current pulses and a recovery time of amplitude and width depending on the electrolyte used. A first pulse deposits the material but charges the barrier layer increasing the thickness of the electrical double layer like a capacitor, which is discharged with a second inverted pulse of the same amplitude of the lowest voltage used to create the dendrites. Then a recovery time, when no potential is applied, is given to replenish the ion concentration and balance the pH around the working electrode so the process can be restarted. As metal ions are reduced at the working electrode by charge transfer and side reactions take place at the counter (Pt) electrode, in all methods the measured (or controlled) current is an indicator of the rate of deposition. The selection of each method depends on the template available, power source, control system, quantity and quality of the deposited products.

TABLE II summarizes some of the most common magnetic materials electroplated into alumina templates. It is not intended to be complete nor to include all references (only one reference per material is used) but to serve as a starting point and as example of the range of possible NW materials that can be

grown using this method. In addition to this, doping can be achieved as for example Pt-doped iron [99] and annealing leads to metal oxides (see for example the end of section III and sections IV and V.A below).

TABLE III
MAGNETIC MATERIALS COMMONLY ELECTROPLATED INTO
ALUMINA TEMPLATES

Material	Common electrolyte reagents	Comment	Reference
Nickel (Ni)	Nickel Sulfate Nickel Chloride	Elemental	[100]
Cobalt (Co)	Cobalt Sulfate Cobalt Chloride	Elemental	[95]
CoNi	Mixture of elemental electrolytes	Alloy	[90]
Co/Ni	Interchange of elemental electrolytes	Multi-segmented	[36]
CoPt	Mixture of elemental electrolytes	Alloy	[101]
Iron (Fe)	Iron Sulphate	Elemental	[102]
Galfenol (FeGa)	Iron Sulfate Gallium Sulfate	Alloy	[45]
Permalloy (FeNi)	Mixture of elemental electrolytes	Alloy	[103]
FeCo	Mixture of elemental electrolytes	Alloy	[104]
FeNiCo	Mixture of elemental electrolytes	Alloy	[105]
FeCoCu	Mixture of elemental electrolytes	Alloy	[106]
FePt	Mixture of elemental electrolytes	Alloy	[107]

In summary, with the described methods, a wide range of NWs with different shapes and intrinsic material properties can be fabricated (**Fig. 4a-d**). Control of the shape anisotropy and demagnetization energy can be achieved by depositing NWs into templates modulated in diameter **Fig. 4c** [64], [91]–[93], [108] and controlling their length either by monitoring the total deposited charge (total length of the NW) or by introducing nonmagnetic segments between magnetic segments and monitoring each segments’ length (**Fig. 4d**) [45], [94], [109]–[112]. Composition modulations can also be performed by selecting the deposited metal ions by manually substituting the electrolyte [36], [90] and it is even possible to match diameter and composition modulations [113].

Fig. 4 SEM imaging of (a) Fe₉₀Co₁₀ NWs with diameter (D) of 60 nm, (b) Fe₃₀Co₇₀ nanotubes with D = 200 nm, (c) Fe₂₈Co₆₇Cu₅ diameter-modulated NWs with D₁ = 120 nm and D₂ = 140 nm, (d) Co/Pt composition-modulated or multisegmented NWs with D = 40 nm.

III. DC APPLICATIONS - PERMANENT NANO-MAGNETS AND ELECTRODES

Whether isolated or collectively, NWs are used as a static source of magnetic fields for different applications. To this end, it is crucial that they are designed with large remanence and coercivity (e.g., high energy product $B \cdot H$) to produce large stray fields and prevent demagnetization, respectively. In highly packed nanowire arrays, the dipolar interaction between neighboring NWs produces an effective demagnetization that reduces the energy product [114], [115]. To overcome this effect the magnetocrystalline anisotropy energy can be tailored to keep the easy axis parallel to the NW's long axis. In this regard, transition metal alloys such as $\text{Co}_x\text{Pt}_{1-x}$ or $\text{Fe}_x\text{Pt}_{1-x}$ NWs constitute a good example, due to their controllable and large magnetocrystalline anisotropy ($K_u \approx 5 \times 10^6 \text{ J/m}^3$) [101], [107]. Alternative magnetic alloys, which do not contain rare-earth elements or Pt, such as FeCo, are also considered, due to their high Curie temperature and large saturation magnetization.

Several attempts have been made to promote a magnetic hardening (increased coercivity) by adjusting the alloy composition, adding a non-magnetic material into the system or by thermal annealing [116]–[119]. As an example of the latter, a significant increase in coercivity and magnetocrystalline anisotropy energy was observed in Fe₃₀Co₇₀ NWs, embedded in alumina templates, by adding 5 at. % Cu, and performing a subsequent thermal treatment. Saturation magnetization, measured under 2 T applied field, resulted in values of about 2.0 T and 1.7 T for as-deposited and 500 °C annealed samples, respectively. After annealing, an increase in coercivity, which increased the $B \cdot H$ energy product, was observed in both, FeCo and FeCoCu samples, but more effectively in the samples containing Cu (**Fig. 5a**).

The large coercivity of thermally treated Cu-doped NWs is attributed to the increase in magnetocrystalline anisotropy energy as a result of *bcc* lattice strain and changes in the NW microstructure [115], [119].

Fig. 5 (a) Low temperature dependence of coercive field of FeCo (orange symbols) and Cu- doped FeCo (black symbols) NWs, with 20 nm in diameter, before and after annealing at 500 °C (figure adapted from [119]), (b) Magnetic hysteresis loops of 40 nm diameter Fe₃₀Co₇₀ NW arrays at 300 K for 3 different states: as-prepared, the top-tips of the NWs covered with FeMn and both (top and bottom) ends covered with FeMn. The inset shows the schematic view of the sandwiched AFM/FM/AFM structure (figure adapted from [104]) © IOP Publishing. Reproduced with permission. All rights reserved).

A different approach for improving the magnetic hardness of NWs consists in exploiting the interfacial exchange between ferromagnetic-antiferromagnetic (FM-AFM) or between ferromagnetic materials with different and high magnetocrystalline anisotropy [120]. As the NWs demagnetize via nucleation from the NW ends or de-pining, any way of stalling this effectively increases the coercive field [104]. One way to achieve this starts by etching the top and bottom layer of alumina, resulting in free-standing FeCo tips sticking out of the template. The free tips are cover by a thin AFM (Fe₅₀Mn₅₀) layer creating an AFM(10 nm)/FM(16 μm)/AFM(10 nm) structure (**Fig. 5b**-inset). The magnetic measurements (**Fig. 5b**) revealed that this sandwiched AFM/FM/AFM structure shows a significant magnetic hardening (50% increase in coercivity and 63% in remanence) with a doubled energy product at room temperature [104].

The stray field emanating from the tips of the NWs in an array depends on their magnetic properties and interactions as well. These interactions are commonly investigated by studying their first order reversal curves (FORC) diagrams [121]–[123] or by directly imaging their fields with TEM tools [12]. For

example, in iron, nickel and cobalt NWs of 50 nm diameter, these stray fields are highly localized and decay rapidly (below 200 nm) with the distance from the tip [12].

Although isolated NWs fabricated inside alumina templates are envisioned to work as MFM tips, to our knowledge, not many reports have appeared, since Yang et al attached single NWs dispersed in water to a cantilever tip using a dielectrophoresis process [15] and Downey et al used a nano-manipulator to pick them from a stripped template [124] mainly to test their mechanical properties (see Fig. 6a).

Fig. 6 Cylindrical nanowires (a) attached to an atomic force microscope tip (Reprinted from [124], with the permission of AIP Publishing.), (b) as vibration sensor (reprinted from [125] © 2013 IEEE), (c) as hematite electrode for water splitting (Reprinted with permission from [126] Copyright 2019 American Chemical Society) and (d) inside a polymer micropillar for force sensing (Reprinted from [127] © 2015 IEEE) (the inset shows the NWs aligned and embedded inside the polymer).

Free standing arrays of bare NWs have been used as cilia for liquids/gas flows [125], acoustic [128] and pressure [129] sensors by placing them in close proximity to the surface of a GMR sensor, as can be seen in Fig. 6b. A limiting factor in those devices is the NW corrosion as well as the fact that the shape anisotropy energy is way larger than the energy changes resulting from the small mechanical deflections. This means that the deflections do not modify greatly the stray magnetic fields, limiting the sensitivity. One way to overcome these limitations is to release the NWs from the array and embed them into a polydimethylsiloxane (PDMS) matrix to create cilia-like structures like the one shown in Fig. 6d [127], [130]. The PDMS structure protects the NWs from corrosion and provides higher mechanical flexibility, allowing more pronounced deflections. This allowed to fabricate force sensors for flow, acoustic, pressure or robotic applications [13], [14], [131]–[133] and energy harvesters [16], [17].

Arrays of NWs and nanotubes have been used as electrodes for multiple applications due to their high surface area. Electrodes of annealed iron, with and without doping, produce

the “hematite” iron-oxide phase known to perform water oxidation in photo-electro-chemical water splitting as shown in Fig. 6c [126], [134]–[136]. On the other hand, several other materials are being grown in alumina templates to produce batteries, supercapacitors and/or fuel cells [137] and have been considered to work as thermoelectric generators [138].

IV. LOW FREQUENCY APPLICATIONS – BIOMEDICINE

Magnetic NWs are being used for various biomedical applications benefiting from the possibility to manipulate them with light [23], [139] or external magnetic fields [22], and sense them with magnetic field sensors (see section III). The applied magnetic field frequencies range from 0.1 Hz external magnetic fields for stem cell scaffolds over 100s of kHz for hyperthermia. When in the presence of air, transition metal nanostructures naturally develop an oxide layer at their surface due to oxygen inward diffusion [26], [140], [141] (See Fig. 7c). This oxide layer makes them biocompatible [142] and biodegradable [143], [144] to the point that they are approved for *in vivo* use in humans [145]. This oxide layer can be thickened by annealing [26] and functionalized to enhance colloidal stability [146], target certain cells [25], [102], [146] and label them [147]. Since the aspect ratio of NWs can be tailored at the fabrication step, they can take shapes from the semi-sphere/cube to the nanowire geometries. High aspect ratio NWs facilitate them to behave as magnetic dipoles with which it is more efficient to perform cell separation with static applied magnetic fields [148], [149].

Ferromagnetic NWs are internalized by living cells as was shown in early *in vitro* studies [150]–[152] without being toxic to the cells (low cytotoxicity) even for high concentrations (~10000 NWs per cell) or long incubation times (5 days). Furthermore, some cells are able to degrade NWs, reducing them to small aggregates and distributing them to neighboring cells [153] possibly due to their polycrystallinity and composite structure [154]. The internalization process takes different times depending on the cells and the NWs, yet once inside, the cells can even divide without problem, as was shown with a machine-learning-assisted sorting of NWs in endosomes [152].

A. Drug delivery

The capability for drug delivery using NWs depends on drug load, cell targeting and controlled release. Low frequency (Hz) external magnetic fields can manipulate single ferromagnetic NWs by producing a torque that induces rotation due to their high aspect ratio. This can be used to propel and push, pull or rotate cellular and sub-cellular objects [155], [156], as shown in Fig. 7a, b. Using a rotating in-plane magnetic field together with a static one perpendicular to the plane, NWs made of Ni and flexible silver have been shown to propel, pick up drug-loaded magnetic microparticles and deliver them through a microchannel to living cells [157]. In addition to this way of loading, antibodies can be coated on the naturally formed oxide layer of NWs (Fig. 7c) by functionalization to perform intracellular delivery (Fig. 7d) [149], [158], [159].

Fig. 7 (a,b) Nickel nanowire (NW) picking, transporting and placing a 6 μm diameter polysilicon microbead on an epidermal cell (Reprinted from [155] Copyright 2012, with permission from Elsevier). (c) Electron energy loss spectroscopy image of an iron nanowire displaying its oxide layer and (d) transmission electron microscopy image of an iron nanowire coated with bovine serum albumin (BSA) (Figures c and d adapted from [158] © 2019 IEEE).

NWs can also serve as remote triggers for other drug-carriers to release the drug. Poly (N-isopropylacrylamide) (PNIPAM) is a micro-gel that swells below a certain temperature and shrinks at a higher temperature, thereby it absorbs and expels a solvent (drug), respectively [160]. When NWs are embedded into PNIPAM, an external magnetic field (kHz frequency range) causes them to vibrate resulting in friction (in addition to possible hysteresis losses) and therefore heating, triggering the release of the solvent. With this method, for example, Rhodamine (B) was loaded into PNIPAM, transported through a microfluidic channel and released in portions into living cells using a pulsed magnetic field of 1 mT and 20 kHz [161]. The ability to release multiple drug doses from the same PNIPAM carrier by repeated application of the magnetic field is an additional feature of this method.

B. Magnetic NWs for cancer treatment

There are mainly three methods (which can be combined) to destroy cancer cells once the NWs have targeted and possibly have been internalized by them: release specific drugs, induce heat or mechanically affect the cell's internal structure. The models for the mechanical heating of magnetic nanoparticles in liquids using external AC magnetic fields have been widely studied [162], [163]. When an AC (kHz) field is applied to NWs internalized by cancer cells, their hysteresis losses makes them act as localized heat sources that induces their death (hyperthermia) [164] as they are especially sensitive to temperature changes [165]. Both the applied magnetic fields

and magnetic properties of NWs play an important role in determining the amount of heat released, which is characterized by the specific absorption rate (SAR) [22]. This SAR can be experimentally quantified by inspecting the hysteresis and minor loops of NWs [166] and relating them with calorimetric studies [167], [168].

Mechanical stress can be induced in living cells using internalized NWs and applying external magnetic fields at low frequencies. Rotating NWs at low frequencies can deform the cytoplasm and cell nucleus [169] or produce cell membrane rupture [4] to cause cell death. For instance, internalized Ni NWs induced cell deaths of fibroblasts in a 240 mT rotating field at 1 Hz [170], colon cancer cells in a 0.5 mT field at 1 Hz [4] or kidney cells at frequencies from around 2 to 11 Hz [171]. These small field and low frequency values make the method particularly attractive from an energy and instrument point of view, which can be kept low and compact, respectively.

Combining several treatment methods into one platform has been shown to increase the efficacy of cancer cell killing. An example of combined drug-release and mechanical actuation on cells was performed with Fe NWs loaded with doxorubicin, targeting breast cancer cells and actuating them with a 1 mT magnetic field at 10 Hz [102]. Recently, those two methods were further combined with photothermal therapy, induced by a near infrared laser. Using an iron/iron oxide core/shell NW, a very high photothermal efficiency of around 80% was achieved and the combinatorial treatment resulted in nearly complete cancer cell eradication [139].

C. Magnetic Resonance Imaging (MRI) contrast agents

In magnetic resonance imaging (MRI) high magnetic fields align, to some extent, the spins of protons from hydrogen atoms (mostly in water molecules). An additional radiofrequency (RF) pulse then excites those aligned spins and the energy released as they re-align with the field is sensed. In different types of tissues, the energy released and the time it takes for the protons' spins to realign (relaxation time) is different, allowing their differentiation. When the static magnetic field is applied, spins precess in phase around it with a frequency known as Larmor frequency, which depends on the magnitude of the applied field. After the RF pulse is applied at 90 or 120 degrees with respect to the static field, the spins relax back to precess around the static field with Larmor frequency in a time T_1 . Nevertheless, some spins remain out of phase longer due to spin-spin interactions, and relax with a higher relaxation time T_2 .

Contrast agents are sometimes used to reduce the relaxation times T_1 and T_2 : the faster they realign, the brighter the image [172]. This is done by creating a local magnetic field, which enhances the proton spins re-alignment, meaning that contrast agents are not directly visualized but they only affect their surroundings. This also explains why most paramagnetic contrast agents shorten T_1 , while superparamagnetic and ferromagnetic shorten mostly T_2 [173]. The performance of a

magnetic nanostructure as a contrast agent is measured via its relaxivity ($r_1=1/T_1$), which is a measure of the relaxation rate and depends on the concentration of the magnetic agent [172]. Then, contrast agents can be used for positive (T_1 , $r_2/r_1 < 5$) or negative (T_2 , $r_2/r_1 > 8$) based images [174] according to their magnetic properties, geometry and coating [175].

Fig. 8 (a) 3D surface reconstruction rendered from a set of T_2 weighted images taken from a mice brain acquired after implantation of NW labeled cells (shown in red). (adapted from [176]). (b) Human mesenchymal stem cell probing the surrounding NW array using filopodia (Republished with permission of IOP Publishing, Ltd from [177]; permission conveyed through Copyright Clearance Center, Inc.). (c) T_2 map of poly-acrylic acid coated nickel NWs used as contrast agent and showing the decrease of relaxation time as the concentration increases (Republished with permission of Royal Society of Chemistry, from [178]; permission conveyed through Copyright Clearance Center, Inc.).

Ferromagnetic NWs with high aspect ratios, which can create large local magnetic field inhomogeneities, can be employed as T_2 contrast agents [179] and have been tested in-vivo as seen in **Fig. 8a**. This has been used, for example, in Fe and Fe-Au NWs, where higher aspect ratios increased r_2 and coating of the Au segments increased r_2 without changing r_1 [27]. Nickel NWs have also been used as T_2 contrast agents at 1.41 T with r_2 values comparable to commercially available contrast agents based on iron-oxide nanoparticles [178]. Their T_2 contrast map measured at 3 T is shown in **Fig. 8c**, where faster relaxation times are found for high concentrations.

D. Cell scaffolds NW

Stem cells research is of growing interest, due to the potential for new treatments of different diseases and conditions. For example, in tissue engineering, mesenchymal stem cells (MSC) can differentiate (change from one cell type to another) into osteoblasts and generate bone tissue, which could assist the

regeneration of patients with bone-degenerative diseases [180], [181]. The properties of the microenvironment and the matrix which the cell is embedded in, were shown to have a direct influence on the cell and its fate, i.e. the type of cell it is going to differentiate into [182], [183]. Consequently, research on stem cell scaffolds has focused on nano-engineering the matrix stiffness and geometry. Studies have been performed, for example, on polymeric nanofibers [184], nano-pits [185], TiO_2 nanotubes [186], hydroxyapatite nanorods [187] and silicon NWs [188].

By releasing magnetic NWs only partly from the alumina template, nano-surfaces densely packed with free standing NWs can be created. Benefiting from the control over geometrical parameters provided by this fabrication method, stem cell scaffolds with different NW diameters can be realized. They mimic different environmental stiffness's, which result in different cell responses. In recent studies, MSCs on ferromagnetic NWs exhibited a contracted shape, shown by the clustering of the focal adhesion vinculin protein [177] (see **Fig. 8b**), which is linked to osteogenesis [188]. An advantage of these ferromagnetic NWs scaffolds over other materials is that they could be manipulated by external magnetic fields, providing interesting opportunities for mechanical cell stimulation on the nanometer scale. On the other hand, arrays of Fe-Au NWs have been used as plasmonic scaffolds to enhance surface enhanced Raman scattering signals [76]. Thereby, small gaps between the NWs is required to obtain a field enhancement effect. NW separations of <10 nm were achieved by widening the pores of the alumina template before electrodeposition.

V. HIGH FREQUENCY - ELECTRONIC ELEMENTS

A. Non-volatile Data Storage

Although arrays of NWs have been proposed to be used as memory devices taking advantage of their high internal interaction field [3], most of the envisioned data storage applications require bits to reside within individual NWs.

Two main types of 3-dimensional magnetic memories using NWs are under research: memristors [189]–[191] and the cylindrical analog of the “racetrack memory” [192]. The former targets NAND flash working in the microseconds (MHz) regime and the latter targets DRAM working at the nanoseconds regime (GHz) [193]. In NWs memristors, one of the most common resistive switching mechanisms consists in applying an electric field which produces point defects migration, creating conductive paths through the oxide layer in a metal/oxide/metal capacitor structure and reducing its resistance (logical 0) [194]. These paths remain after the electric field that created them has been removed and shrink with the application of heat or another electric field of inverse polarity, returning the structure to a high resistance state (logical 1). This type of memory has been reported in homogeneous NiO [195], multisegmented Au/NiO/Au [196],

Pt/NiO/Pt [197], Ni/CuO_x/Ni [198], Ni/NiO/Ni [199], core-shell Ni/NiO [200] and TiN/TiO₂/Pt [201] NWs amongst others. A thorough compilation of structures and measurement techniques can be found in [190].

Due to their high shape anisotropy, NWs typically represent one dimensional magnets with their magnetization pointing parallel or antiparallel to their long axis (up or down in **Fig. 9a**). This could be exploited for data storage, where parallel magnetization corresponds to logic 0 and antiparallel to logical 1. In order to realize a multi-bit 3D memory, several bits need to be stored along the NWs, as shown in **Fig. 9b**, requiring multi-domain states. This can be achieved by creating DW pinning sites along multisegmented NWs via compositional changes [202] or diameter modulations [37], [203]. Each segment's magnetization direction reverses either by coherent rotation or by nucleation and propagation of a DW, depending on their composition (e.g., defining its magnetocrystalline anisotropy) and length [111], [204]. This reversal can be driven by magnetic fields [36], [37], electric currents [43], [205] or a combination of both [44] with DWs possibly transforming during motion [206]. In particular, the experiments with cobalt and nickel-based NWs of diameters around 90 nm have resulted in current-induced DW velocities up to 600 m/s [44], [205].

Fig. 9 Magnetic memory designs based on cylindrical NWs. (a) Perpendicular anisotropy magnetic tunnel junction memory: one bit can be stored in the magnetization state of each junction. (b) Multisegmented NWs with several bits stored in the magnetization state of each segment.

One of the main challenges of these perpendicular anisotropy memories is the stray field of each memory element that creates interference as the element density increases and they are packed closer together. This effect has been widely studied for NWs [103], [108], [122], [207], [208] and could now be useful for perpendicular anisotropy magnetic tunnel junction (p-MTJ) based memories [209], [210] as their packing density increases. In these p-MTJs, a thick ferromagnetic (storage) layer is deposited on top of an MTJ so that its thickness is in the order of or larger than the diameter of the MTJ [209], [211] or the thickness of the free layer of a MTJ is increased until the shape anisotropy dominates [210]. This combination of perpendicular

shape anisotropy and interfacial perpendicular magnetic anisotropy increases the device's thermal stability, i.e., the minimum energy required to switch the memory between two states well above room temperature activation. A cylindrical 3D "racetrack memory" could be considered an extension of p-MTJs, which are used in magnetic random access memories (MRAM) [209], [210], and are produced by companies like Toshiba, Samsung and Everspin-Global Foundries, among others [193].

B. Microwave electronics

Arrays of ferromagnetic NWs absorb microwaves at tunable resonant frequencies depending on the deposited magnetic material (or materials), their geometry, membrane porosity and an optional applied external field [212]–[214]. This absorption occurs during the ferromagnetic resonance (FMR) when the applied microwave frequencies coincide with the frequency at which magnetic moments precess about the internal effective magnetic field. This occurs dynamically: the microwave magnetic field knocks the magnetic moments away from the internal effective magnetic field and they precess about this latter as they re-align. Resonance occurs, then, when the microwave magnetic field reinforces the precession at the same frequency. The internal effective magnetic field depends on the magnetic energy terms, which can be tailored, as discussed in section II, namely exchange, crystal and shape anisotropy and magnetostatic interactions. Through this mechanism, most of the microwave is absorbed by the NWs, which then act as tunable filters.

A common approach to fabricate microwave devices is using the NWs embedded in the template as a substrate in a microstrip as can be seen in **Fig. 10a**. This microstrip consists of a conducting line separated by the substrate from a ground plane, the whole of it being a transmission line. In addition of being a dielectric, this substrate has magnetic properties which are appreciable thanks to the NWs diameters being smaller than their skin depth at high frequencies [212]. These magnetic properties contribute to the majority of the losses when a microwave is passed through the transmission line due to the FMR explained before [212], [214]–[216]. Vector network analyzers are used to generate and route these microwaves into the NWs microstrip through (commonly) two ports and characterize their scattering parameters (S). These scattering parameters are related to both the amplitude and phase shift (ϕ) properties of the scattered waves at the microstrip and quantify the power losses.

The frequency response of the transmission scattering coefficient S_{21} (forward from port 1 to port 2) for CoFe NWs, both measured (black) and simulated (gray), is shown in **Fig. 10b** [214]. The strong absorption dips are related to the FMR and are present even at zero applied field, as explained before. The DC magnetic field applied along the long axis of the NWs modifies the precession frequency of the magnetic moments as they re-align increasing the frequency needed to reach

resonance.

Clever, slight modifications to the growth methods explained in section II.B allow the production of microstrips where the NWs height changes along the conducting line width [217], [218]. In other words, the effective permittivity of the substrate varies due to the change in absorption depth along the width. This asymmetry causes the microstrip to behave differently in forward (+) and backward (-) (from port 2 to port 1) wave propagation directions. Consequently, the difference in phase shifts of the forward and backward propagation directions, $\Delta\varphi = \varphi_+ - \varphi_-$ differs from zero, as seen in **Fig. 10c** for different values of applied field turning this microstrip into a phase shifter device.

Fig. 10 (a) NWs inside the template as a substrate of a microstrip. (b) Transmission coefficient S_{21} of CoFe NWs with a DC magnetic field of 2, 4 and 6 kOe applied parallel to the wire axis (Reprinted from [214] Copyright 2008 with permission from Elsevier). (c) Differential phase shift ($\Delta\varphi$) per total length of the microstrip L at different applied DC magnetic fields from 2 to 8 kOe (Reprinted (adapted) with permission from [218] Copyright 2010 American Chemical Society).

In general, a proper design of the transmission line geometry [219], [220] and substrate's dielectric and magnetic properties [212], [214]–[216] allows the fabrication of circulators [29], [31], filters [221] and phase shifters [32], [218] with a wide frequency range.

VI. SUMMARY, OUTLOOK AND CHALLENGES

The electrochemical deposition of cylindrical magnetic nanowires into alumina templates is a fabrication method that features precision in terms of NW geometry, control over the NW material, batch fabrication and freedom over various design parameters. As a consequence, it has been utilized in many studies of fundamental properties as well as to implement multiple applications. In this review, we covered the basics of this versatile fabrication method and summarized the various materials and designs that have been realized, leading to different means to tailor the NW properties. Then, we covered the applications of these NWs, starting with the permanent magnetic applications over the low frequency regime, which includes biomedical applications, to high frequency operations as memory elements or electronic components.

The review of these applications revealed that NWs excel in many ways over other implementations, thanks to their unique features. The translation of these research results into commercial products, however, has yet to follow. One obstacle that most of these prospective devices share with many others is that their fabrication process has not been fully integrated into standard Si processing methods, despite efforts in anodizing thin sputtered or evaporated layers of aluminum on prevalent industry substrates (see for example section 7.5 in [48]). Therefore, industrial-scale manufacturers of NWs and membranes with highly reproducible and precise properties are scarce. On the other hand, released NWs are not widely commercially available and are employed in small quantities. Compared to, for example, the high production yields of superparamagnetic nanoparticles, which do not necessarily require a template, the low yield of NWs in large lots limits their widespread application and utilization and imposes a hurdle to enter the research field.

One aspect that needs to be further developed is single crystallinity of NWs. This has not been attained consistently and could allow huge improvements in many applications, especially of NWs used as electrodes, where defects limit charge carrier diffusion lengths. A tighter control over the electroplating conditions could help in this regard too: most research groups work with setups built in-house with variable solution quantities, temperature, stirring and atmospheric conditions. The same issues are faced when fabricating alumina templates using anodization and higher degree of pore-order etc. are desired.

Attempts to use arrays of nanowires as an alternative family of permanent magnets without rare-earth elements have been partly successful in spite of their relevance for energy-related

applications. Their relevance, arises from the possibility to superimpose the magnetocrystalline or magnetoelastic anisotropies to the geometrical shape anisotropy resulting in stronger total axial magnetic anisotropy, and hence, stronger, tunable magnets. Nevertheless, this total magnetic anisotropy is still low compared to the anisotropy found in rare-earth containing alloys (e.g., FeNdB) which results in their high magnetic coercivity. However, this last one can be enhanced by pinning the domain wall which reverses the NW's magnetization by suitable geometrical (e.g., by sharpening the ends) or compositional (e.g., by growing novel alloys with particular properties as high Curie temperature) designs. In any case, higher packing density of nanowires is needed in order to reach larger magnetization values. This unfortunately introduces significant magnetostatic interactions between the NWs reducing the effective axial anisotropy of the whole nanowire array.

In the area of biomedical applications magnetic NWs are a promising approach toward a practical solution for nano-robots. They offer position control, visualization and multi-modal actuation (vibration, rotation, heating) remotely and enable drug deliver as well as functionalization for specific targeting in a small, customizable package. Combining those capabilities into a single nano platform can result in powerful tools for theranostics or personalized medicine, which has been partly shown in some works already. However, more research is needed to optimize these instruments and better understand the many ways of interaction between the NWs and cells. An inevitable step in the future has to be in-vivo testing of the NWs and long-term studies. A concern that needs to be better addressed is agglomeration of the NWs, due to magnetostatic interactions. Hence, surface coatings need to be further optimized to aid their dispersion and selectivity.

On the other end of the frequency range, 3-dimensional magnetic memories and FMR-based microwave electronics are very promising. They benefit from the template fabrication method, which provides out-of-plane magnetic structures separated by an electrical insulation layer with aspect ratios that cannot be realized by standard Si fabrication technologies. A critical factor for further development of these devices will be the successful integration with standard electronics on-chip. In case of microwave applications of cylindrical magnetic NWs, the field dependence of resonant frequencies could be exploited for sensing applications, which could be especially attractive for wireless sensor networks.

Through the large range of materials and properties that have been developed for cylindrical magnetic NWs, fascinating opportunities have been created, and the number of applications keeps growing. A further impact and technological translation of these NW-based solutions could be obtained by implementing a scaled-up fabrication process that would provide better accessibility to this technology.

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