Tuning shape, composition and magnetization of three-dimensional cobalt nanowires grown by Focused Electron Beam Induced Deposition (FEBID)

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Abstract

Electron beam induced deposition of three-dimensional cobalt nanowires with simultaneous high metallic content (≈80 % at.) and small diameter (< 100 nm) has been achieved by optimization of the growth parameters. Two different growth modes have been identified, denoted as *radial* and *linear*. In the *radial* mode, the wire diameter is at least ≈120 nm and the Co content is greater than ≈85% at.. In the *linear* mode, the diameter is smaller than 80 nm and the Co content is at best

≈80% at.. A sharp transition between both growth modes can occur inside a single nanowire for certain experimental conditions. Electron holography measurements indicate that in optimized Co nanowires the magnetic induction is high enough for applications in spintronics and magnetic sensing and actuation at the nanoscale.

Keywords: cobalt nanowires, focused electron beam induced deposition, electron holography, magnetic nanowires

1. Introduction

Thin-film layers and multilayers based on magnetic materials have nowadays various applications in the fields of sensors and data storage, like in hard disks [1] [2]. On the other hand, individual magnetic nanostructures are being investigated for their potential application in sensors [3], memories [4] and logic [5]. Although most of the approaches for their fabrication rely on standard lithography processes performed onto magnetic thin films and multilayers, a growing interest exists on three-dimensional magnetic nanostructures, whose fabrication is challenging. Focused Electron Beam Induced Deposition (FEBID) is one of the techniques that allow addressing the growth of three-dimensional structures [6] [7] [8] [9] [10], in particular those based on magnetic materials [11] [12] [13] [14] [15] [16] [17] [18] [19] [20]. In FEBID, precursor molecules delivered by a Gas Injection System (GIS) close to the substrate become dissociated by a focused electron beam, producing a deposit [21] [22] [23] [24]. The shape of the deposit is determined by the electron beam scan as well as complex interactions between the electron beam, substrate, precursor molecules and growing structure [25] [26]. The use of precursor molecules containing magnetic elements such as Co, Fe and Ni permits the growth of magnetic deposits [11] [27] [28] [29] [30] [31] [32] [33] and a large development has been made towards the growth of magnetic deposits with high metal content, high magnetization, high resolution and complex shapes, as recently reviewed [34] [35]. Such development has been focused on the optimization of thin in-plane magnetic layers, whereas limited work has been done with regard to three-dimensional magnetic deposits. However, there are many promising applications of three-dimensional magnetic deposits in scanning probe techniques (such as Magnetic Force Microscopy [17] and Ferromagnetic Resonance Force Microscopy [36]), racetrack-type magnetic memories [14], Hall sensors [37] [38], nano-magnet logic [17] [39], superconducting vortex lattice pinning [40], remote magneto-mechanical actuation [20], etc.

In the present work, we investigate in detail the interplay of the precursor flux and the electron beam current in the physical properties of out-of-plane magnetic nanowires grown by FEBID using the $Co_2(CO)_8$ precursor. Our focus is put on the characterization of the obtained nanowire's diameter, composition and magnetization, with the aim of growing narrow nanowires (< 100 nm in diameter), with high Co content (> 80% at.) and magnetization approaching the bulk value (1.8 T).

Previous work on the growth of three-dimensional nanowires by FEBID has shown the relevance of several parameters that should be taken into account. For example, the use of sub-nA electron beam currents produced by field-emission guns is suitable for the growth of narrow nanowires (< 100 nm in diameter) [14] [41]. Additionally, the interaction of the primary electron beam with the substrate and the growing structure also depends on the primary electron beam energy [42] [43]. The balance between the availability of precursor molecules on the growth area and the electron beam current is very important because it will determine whether the growth occurs in the precursor-limited regime or the electron-limited regime, which will affect not only the growth rate but also the composition of the nanowire [44]. However, this equilibrium can be strongly modified when thermal heating of the growing deposit occurs, as previously found in FEBID [45] [46] [47] [48]. On the one hand, an increase of temperature in the area of growth will change the precursor residence time, affecting the growth rate and potentially the growth regime. On the other hand, the decomposition of the precursor molecules will be faster if temperatures close to the thermal decomposition of the precursor are reached. Moreover, thermal effects can be of tremendous importance in three-dimensional nanostructures given that precursor replenishment in the area of growth occurs at a lower rate compared to in-plane deposits because the diffusion mechanism of precursor molecules from the substrate will be weakened as the deposit grows in height. In fact, our results presented hereafter have identified a set of growth parameters that produce a change in the diameter during the growth of a single nanowire. This is a consequence of the subtle balance between the different factors governing the growth of three-dimensional nanowires, as discussed hereafter.

2. Experimental

2.1 Growth of the three-dimensional nanowires by FEBID

The nanowires were grown in commercial Helios Nanolab 600 and 650 Dual Beam equipment using the Schottky field-emission electron gun and a GIS that delivers the $Co_2(CO)_8$ precursor. The substrates were naturally-oxidized Si wafers. FEBID-Co deposits were grown with low electron beam currents (< 100 pA). The working voltage was fixed to 5 kV given that initial experiments did not lead to significant changes in the composition from 5 kV to 30 kV. The nanowires were grown in spot mode, where the electron beam is continuously irradiating a single point. A base pressure of $1x10^{-6}$ mbar existed in the working chamber before the injection of the precursor. The $Co_2(CO)_8$ precursor flux was tuned via a manual valve, which permits to vary the chamber pressure during growth up to $\sim 4\times10^{-5}$ mbar. Given the linear relationship between the chamber pressure increase during gas injection (ΔP) and the precursor flux (J), $J \alpha \Delta P$ [49], monitorization of the chamber pressure during growth allows us to establish relative correlations.

2.2 Compositional analysis by Energy Dispersive X-ray Spectroscopy (EDS)

Some of the EDS experiments were performed inside Helios Nanolab 650 Dual Beam equipment, using an excitation electron beam voltage of 5 kV, beam current of 800 pA, and analyzed with the EDAX software using APOLLO X detector. Other EDS experiments were carried out inside an FEI Tecnai F30 Transmission Electron Microscopy (TEM) equipment operated at 300 kV. In this case, the used software was Genesis RTEM, which is a tool embedded in FEI's TIA software package, using a 136-5 detector from EDAX. The material composition was determined through these experiments with a typical error of ~2% at. for main components assuming uniform composition in the nanostructure. Along the manuscript, the composition is always expressed in at. %.

2.3 Compositional analysis by Energy Electron Loss Spectroscopy (EELS)

EELS experiments were performed in an FEI Tecnai F30 equipment and in a probe-corrected Titan Low Base 60-300 equipment, both operated at 300 kV. The first one is fitted with the Tridiem 863 Gatan Energy Filter (GIF) whereas the second one is equipped with a high brightness Schottky electron gun (S-FEG), a CETCOR corrector for the condenser system to provide sub-Angstrom probe size, and a Tridiem GIF (866 ERS). The spectroscopic experiments were carried out with a

25 mrad convergence semi-angle and EELS spectra were performed with an energy dispersion of 0.8 eV and energy resolution around 1.5 eV.

2.4 Analysis of the magnetic induction by Electron Holography (EH) inside a Transmission Electron Microscope (TEM)

EH was carried out in an image-corrected FEI Titan Cube 60-300 TEM equipment operated at 300 kV and equipped with an S-FEG and a CETCOR corrector for the objective lens and a motorized electrostatic biprism. The experiments were performed in Lorentz mode (with the objective lens switched off, and the Lorentz lens, fitted below the objective lens, operating as the image-forming lens). In the holographic experiments, the excitation of the biprism was varied between 180 and 220 V, depending on the actual diameter of the NWs, to produce holograms with a fringe contrast range of 20-25%. The acquisition time of the holograms was set to 5 s. The method to extract the magnetic induction has been described in detail in a previous publication [41].

3. Results

As previously mentioned, a low electron beam current is a pre-requisite for the growth of small-diameter nanowires. First, we present the results obtained using an electron beam current of 86 pA. As can be observed in Figure 1(a), a narrow nanowire with diameter of 62 nm and an aspect ratio of 25 is obtained when ΔP is 7.3 x 10^{-6} mbar. However, a decrease in ΔP to 6.4 x 10^{-6} mbar provokes a change in the growth mode at the height of 650 nm, resulting in a nanowire with a small diameter in the first segment (66 nm) and larger diameter in the second one (119 nm), as shown in Figure 1(b). A further decrease in ΔP to 5.9 x 10^{-6} mbar induces the appearance of the larger diameter closer to the substrate, at the height of 160 nm (see Figure 1(c)). If an even lower ΔP of 5.1 x 10^{-6} mbar is used, the nanowire grows from the beginning in the mode with larger diameter, 120 nm, as shown in Figure 1(d). Hereafter, the growth mode with smaller diameter is referred as "linear regime" whereas the growth mode with larger diameter is referred as "radial regime". It is experimentally observed that if the growth current is increased, the radial-to-linear crossover occurs at higher precursor flux (chamber growth pressure). A quantitative model to explain this change in the mode of growth is beyond the scope of the present article given its complexity, but is being currently addressed by the authors. Thermal and/or diffusion effects are expected to play a crucial role in the observed effect. Thus, an increased thermal desorption of the precursor [50] will occur due to an increased temperature at the tip of the nanowire due to reduced thermal dissipation at long wire lengths. Additionally, a reduced number of molecules will be able to diffuse from the substrate as the nanowire grows.

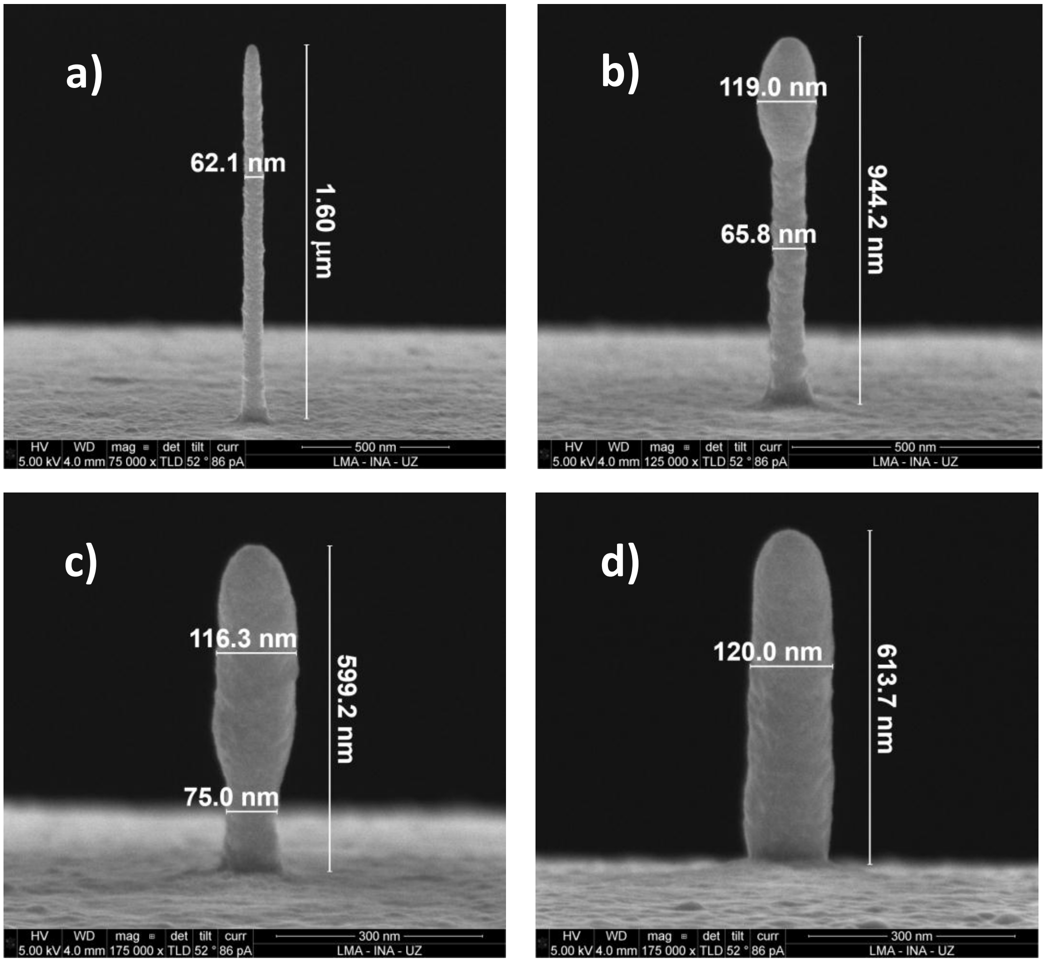

Figure 1. SEM images of cobalt nanowires grown under the following conditions: 5 kV, 86 pA and working pressure (minus base pressure) of (a) 7.3 x 10^{-6} mbar, (b) 6.4 x 10^{-6} mbar, (c) 5.9 x 10^{-6} mbar, and (d) 5.1 x 10^{-6} mbar. The transition from linear to radial growth mode with decreasing precursor flux is noticed.

Similarly to the case of in-plane deposits, the height growth rate of the obtained nanowires increases with the working pressure, as shown in Figure 2, which is indicative of growth in the precursor-limited regime [44]. However, as noticed in this figure, a change in the growth-rate slope is observed at the crossover between the linear and radial growth modes, highlighted with two visual guide lines. It should be stressed that the average height growth rate is well defined for nanowires with pure linear or radial growth modes but, in the case of nanowires with transition between both modes, this value will depend on the relative contribution of both segments to the total height. The height growth rate was determined from data in Table 1 considering the total height of the nanowire and the deposition time, defined as the time spent to grow it. Additionally, the volume growth rate as a function of the working pressure was calculated, increasing linearly in the linear growth mode.

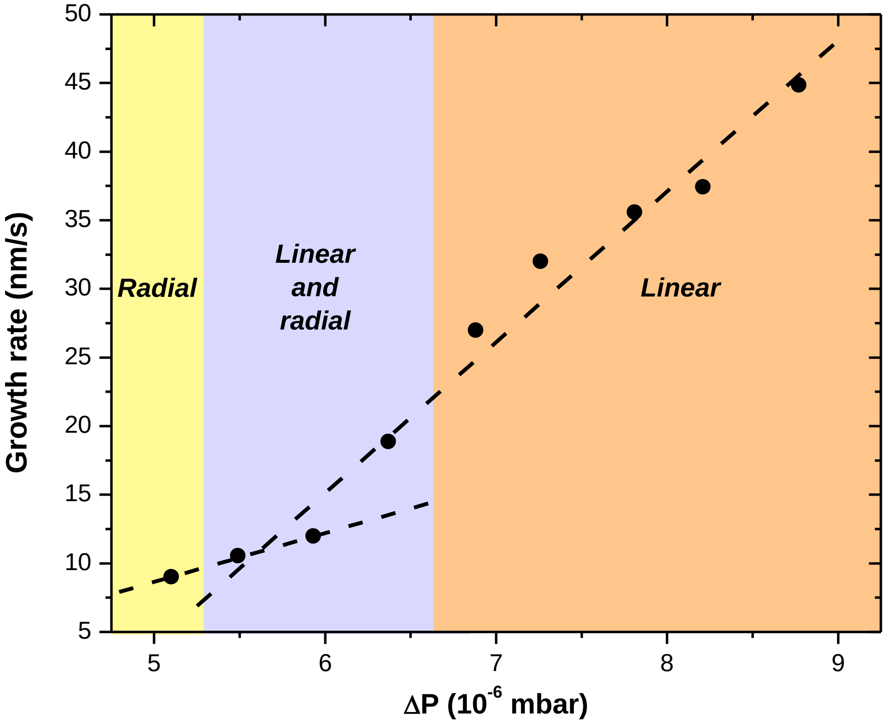

Figure 2. Growth rate of nanowires grown at 5 kV and 86 pA as a function of the working pressure (minus base pressure). A change in slope is noticed at the crossover from radial to linear growth modes. In the "linear and radial" growth mode, the nanowire presents two segments, one with the features of the linear mode and one with the features of the radial mode.

Table 1. Data of the nanowires represented in Figure 2: height, deposition time, growth rate and working pressure (minus base pressure) during growth.

Height (µm)	Deposition time (s)	Growth rate (nm/s)	ΔP (10^{-6} mbar)
1.66	37	44.9	8.8
1.61	43	37.4	8.2
1.53	43	35.6	7.8

1.60	50	32.0	7.3
1.35	50	27.0	6.9
0.944	50	18.9	6.4
0.599	50	12.0	5.9
0.655	62	10.6	5.5
0.614	68	9.0	5.1

As shown in Figure 3, the composition of the deposits is strongly affected by the precursor flux. A dedicated experiment was performed in which the electron beam current was fixed to 100 pA. At that beam current, the crossover from the radial-growth regime to the linear-growth regime occurs at ΔP of 1.75×10^{-5} mbar. Overall, the behavior of the Co content as a function of working pressure resembles that observed in in-plane deposits [38]: an optimum precursor flux window (1×10^{-5} mbar $< \Delta P <$ 1.5×10^{-5} mbar) exists where the Co content is high (~85%). Although specific experiments and/or simulations could shed more light on the origin of this change in composition, from general arguments it can be stated that at lower precursor flux the Co content can diminish due to decomposition of residual contaminant species in the chamber, whereas at higher precursor flux the Co content can diminish due to incomplete precursor decomposition. The different origin of the decreased Co content at low and high precursor flux can be also noted in the C/O ratio, which is smaller than 1 at high precursor flux and larger than 1 at low precursor flux (see Figure 3). From Figure 3, it is clear that optimum Co content (> 85%) can be only achieved in the radial-growth mode, where the diameter is at least ≈120 nm.

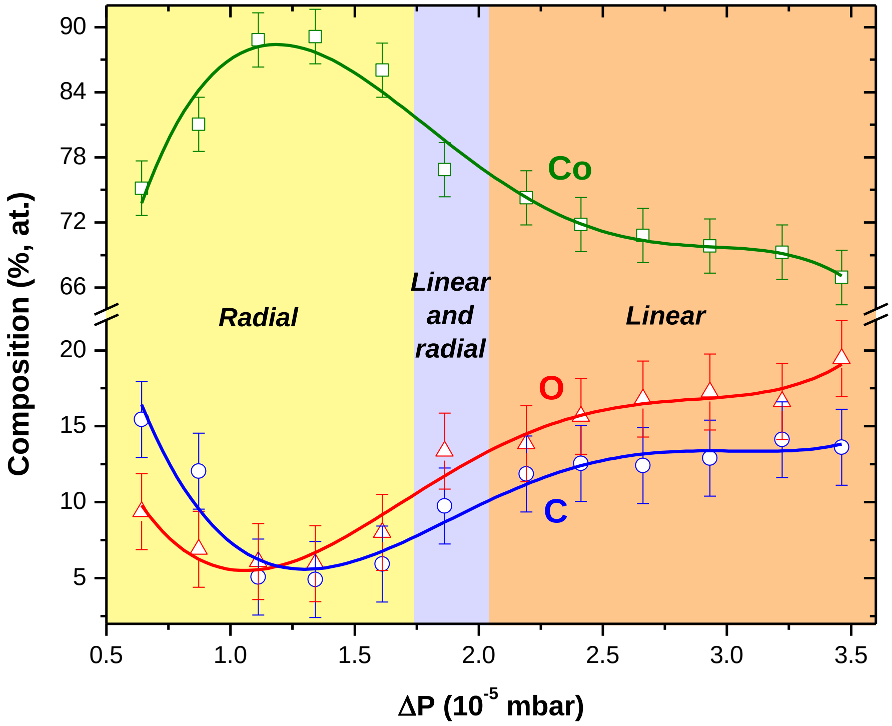

Figure 3. Composition of NWs grown using 5 kV and 100 pA as a function of the working pressure (minus base pressure). EDS measurements were performed at 5 kV and 800 pA. Three different growth regimes can be noticed. The composition of the sample falling in the "linear and radial" regime has been determined at the base of the nanowire, which corresponds to the linear-growth mode.

In order to correlate the Co content of the nanowires with their magnetization, dedicated experiments have been carried out inside the TEM. The experiment consists of EDS of all nanowires and EELS of two selected nanowires. EH has also been performed on selected individual nanowires to obtain quantitative values of the Co content and the magnetic induction inside the nanowire. In Figure 4, the Co content is represented as a function of the nanowire's diameter for optimum growth conditions. The specific growth parameters of each nanowire displayed in Figure 4 are described in Table 2. Figure 4 indicates that a high Co content (> 85%) can be achieved in nanowires with diameter larger than ≈120 nm, which correspond to the radial-growth mode. However, the Co content in nanowires with diameter smaller than ≈80 nm, which correspond to the linear-growth mode, is around 80% for diameters of ≈80 nm, but diminishes quickly as the diameter is reduced. For diameters of ≈60 nm, the Co content is only ~45%. Given that the nanowires present typical oxidized shells of around 5 nm [41], the measured average Co content will be lower as the wire diameter decreases. This means that in the core of the nanowire the Co content is expected to be higher than the average value, this effect being more significant for the narrowest wires.

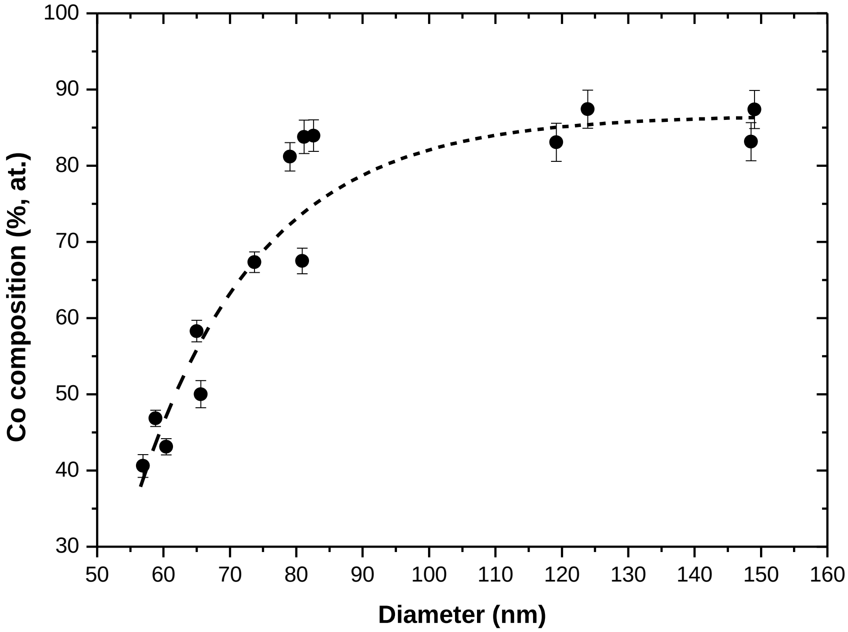

Figure 4. Cobalt composition as a function of the wire diameter for optimized growth conditions at each particular value of the diameter.

Table 2. Data of the nanowires represented in Figure 4: diameter, Co content and technique used for its measurement, beam current and working pressure (minus base pressure) during growth. The beam energy used for the growth was 5 kV.

Diameter (nm)	Co composition (%, at.)	Technique	Growth current (pA)	ΔP (10^{-6} mbar)
56.9	40.6	EDS	50	2.8
58.8	46.9	EDS	25	10.3
60.4	43.1	EDS	50	10.2
65.0	58.3	EDS	100	10.5
65.6	50.0	EDS	100	9.3
73.7	67.3	EDS	100	8.1
79.1	81.2	EDS	50	6.9
80.9	67.5	EDS	100	8.1
81.2	83.8	EDS	100	8.4
82.6	84.0	EDS	50	7.8
119.2	83.1	EELS	50	2.8
123.9	87.4	EDS	50	5.2
148.5	83.2	EDS	100	6.9
149.0	87.4	EELS	100	6.1

The magnetic induction of selected nanowires has been investigated by means of EH. Each nanowire is measured in magnetic remanence after previously saturating the magnetization in two opposite directions by applying an external magnetic field produced by the objective lens. This is a common method to get rid of the electrostatic contribution to the phase change and to reveal the magnetic contribution after subtraction of both measurements. Following the electron holography method described in previous work [41], the average magnetic induction inside a nanowire along its long axis, B_x, can be calculated as:

$$\left|B_x(x,y)\right| = \frac{\hbar}{e \cdot t}\frac{\partial \varphi_{MAG}(x,y)}{\partial y} \tag{1}$$

where $\hbar$ is the reduced Planck constant, φ_{MAG} the magnetic component of the total electron phase shift $\varphi(\vec{r})$, e the electron charge and t the variable thickness along the specimen width. In Figure 5, the results corresponding to three nanowires, representative of the three regimes found in this study, are shown. The values obtained for B_x close to the nanowire borders are not reliable due to the uncertainties in the sample thickness at those positions and edge effects at the oxidized wire surface. For this reason, in Figure 5 the values of B_x obtained at the edges of the nanowires are masked with a semi-transparent band. However, the values obtained in the central part of the nanowires are trustworthy. The nanowire with the largest diameter, 123.9 nm, corresponding to the radial-growth mode, presents a high magnetic induction along the long wire axis of ~1.33 T, not far from the bulk value, 1.8 T. This high value of the magnetization correlates well with the high Co content in the nanowire, 87.4%. A second nanowire, corresponding to the intermediate linear-radial-growth mode has been analyzed by EH at the base, in the portion with linear-growth mode. It presents a magnetic induction along the long wire axis of 0.78 T, around 50% of the bulk magnetization of Co. This reduction is expected given the reduced Co content (67.5%) in this nanowire. A third nanowire, corresponding to the linear-growth mode, presents a lower magnetic induction along the long wire axis of 0.41 T, which can be expected given its reduced Co content (40.6%). However, we would like to point out that the obtained magnetic induction in the nanowires is sufficiently high for functional nanomagnetic devices and applications. Just as an example, the Fe magnetic rods used in the past by Franken et al. had magnetization of 0.13 T along their long axis and were able to pin domain walls in a domain-wall conduit [16].

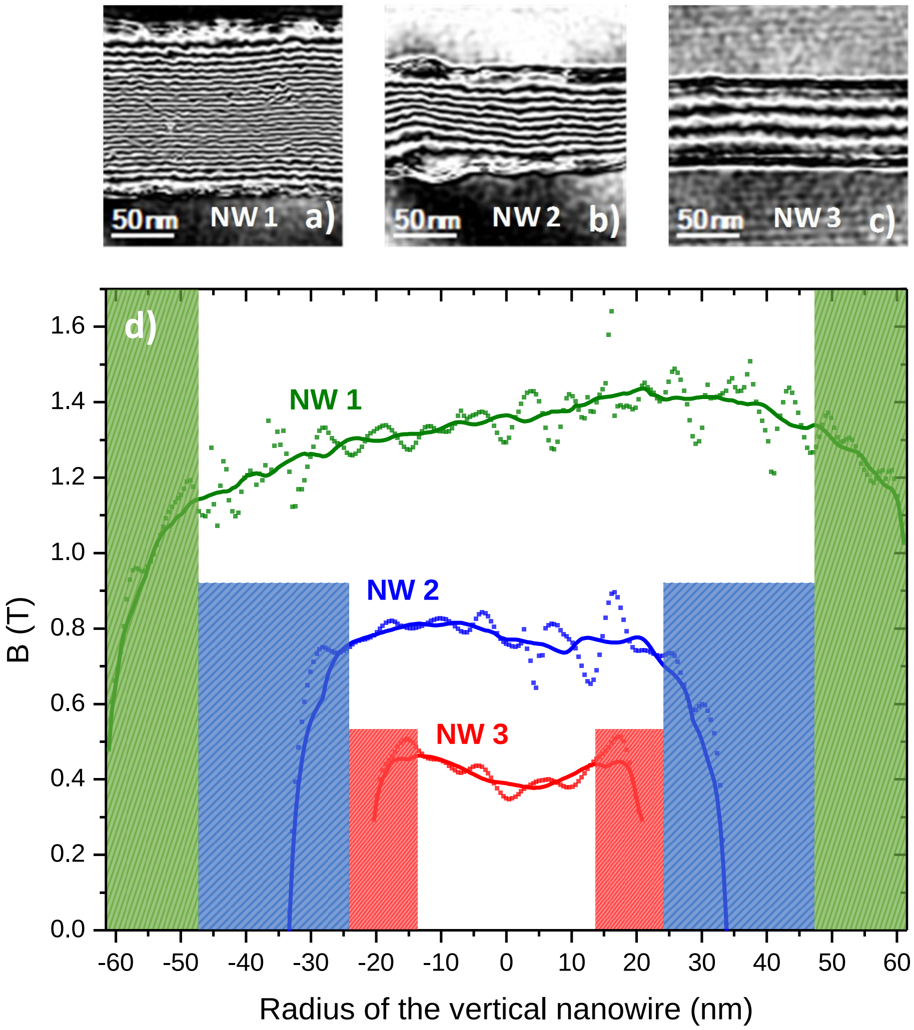

Figure 5. a) Representation of the magnetic induction flux of a nanowire with cobalt content of 87.4% at. (NW1), obtained from the magnetic phase images after normalizing by the maximum thickness and performing the cosine of 700 times the change in electron phase; b) the same for the nanowire with cobalt content of 67.5% at. (NW2); c) the same for the nanowire with cobalt content of 40.6% at. (NW3); d) profiles of the magnetic induction along the short axis of nanowires NW1, NW2 and NW3, obtained from the magnetic phase images used to calculate a), b) and c), respectively. The edges of the nanowires are partially masked given that quantification is not reliable due to edge effects.

4. Discussion

FEBID growth of functional magnetic nanostructures requires precise control of a high number of growth parameters. Their precise tuning can be crucial in particular cases, such as the growth of Co three-dimensional nanowires discussed in the present work. In the process of optimization of their growth, we have encountered a number of interesting phenomena that should be taken into account for their practical application. The first important finding regards the existence of two growth modes with different physical properties, denoted linear and radial due to certain similarities with reported growth of three-dimensional iron nanowires [45]. In the radial-growth mode, the minimum diameter obtained is ≈120 nm and the Co content can be very high, > 85%, showing a high magnetization, not far from the bulk value, 1.8 T. In the linear-growth mode, the diameter can be lower than ≈80 nm and the Co content diminishes for decreasing diameter. For a diameter of 80 nm, nanowires can attain Co content of 80% and show magnetization around half the bulk value. However, if the diameter is 60 nm, the Co content is found to be 45% and the magnetization is around 1/4 of the bulk value. We cannot discard that the nanowires of low Co content have areas with inhomogeneous composition, the areas richer in Co contributing more to the magnetization of the nanowire. Interestingly, inside the same nanowire, a transition between both growth modes can be observed in a certain range of growth parameters. This effect seems to indicate that thermal desorption and decreased diffusion effects during the growth of high-aspect-ratio 3D nanostructures may be playing a key role. The capacity to dissipate the heat caused by the electron beam is reduced as the nanowire grows, being the tip growing progressively further away from the substrate. At a certain height, there is an overheating which could result in a change of the growth mode. The existence of single nanowires with two diameters seems useful for studies of magnetic-domain-wall propagation in nanowires, given their tendency to become pinned at the location of the transition between both diameters [51].

The correlation found between the diameter of the nanowire and its composition is important given the relationship observed between the Co content and the magnetization of the nanowire. If a nanowire with magnetization close to the bulk value, 1.8 T, is required, the best option is to grow a nanowire with diameter of at least 120 nm. However, in many practical situations, narrow nanowires (< 100 nm) are required, in which case, a maximum Co content of ~80% can be achieved, this value diminishing strongly with decreasing diameter. In such situation, the magnetization is observed to decrease with respect to the bulk value despite being still quite large in absolute value. There are a few potential applications of these nanowires such as magnetic functionalization of cantilevers [52] [11] [17] [53] [13], three-dimensional logic structures [17] [39], cylindrical conduits for domain-wall propagation [14], superconducting vortex lattice pinning [40] [54], remote magnetomechanical actuation [20], etc. where lateral resolution is more important than the absolute value of the magnetization. In those cases, the type of nanowires grown here in the linear-growth mode meet the required physical properties. Another strategy to enhance the Co content is to perform post-annealing treatments [55] [56]. It has been shown that in-plane Co structures can be purified by annealing in vacuum conditions, eliminating the oxygen content of the deposits [56]. This could be a viable strategy to obtain narrow Co nanowires (< 100 nm in diameter) with very high Co content (>90%) and the associated magnetization close to the bulk value.

5. Conclusions

To conclude, we have shown that control of the growth parameters in focused-electron-beam-induced deposition, especially the electron beam current and the precursor flux, allows tuning the diameter, composition and magnetization of three-dimensional cobalt nanowires, grown using the $Co_2(CO)_8$ precursor. A transition between two growth modes, radial and linear, has been unveiled in single nanowires, resulting in individual nanowires with two different diameters (80 nm and 120 nm respectively). The best growth conditions to achieve nanowires with small diameter (< 80 nm), high metallic content (~80%) and high magnetization (~0.9 T) have been identified, providing a growth route for various applications.

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