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Magnetic properties of cobalt microwires measured by piezoresistive cantilever magnetometry

Abstract: We present the magnetic characterization of cobalt wires grown by focused electron beam-induced deposition (FEBID) and studied using static piezoresistive cantilever magnetometry. We have used previously developed high force sensitive submicron-thick silicon piezoresistive cantilevers. High quality polycrystalline cobalt microwires have been grown by FEBID onto the free end of the cantilevers using dual beam equipment. In the presence of an external magnetic field, the magnetic cobalt wires become magnetized, which leads to the magnetic field dependent static deflection of the cantilevers. We show that the piezoresistive signal from the cantilevers, corresponding to a maximum force of about 1 nN, can be measured as a function of the applied magnetic field with a good signal to noise ratio at room temperature. The results highlight the flexibility of the FEBID technique for the growth of magnetic structures on specific substrates, in this case piezoresistive cantilevers.

Keywords: Piezoresistive cantilever magnetometry, cobalt focused electron beam induced deposition

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1 Introduction

Magnetic nanowires and nanostripes are valuable candidates for the development of applications such as high-density and high-speed magnetic information storage, magnetic random access memories, magnetic sensors and logic devices [1]. These nanostructures have a different magnetic behavior as a function of their shape, size, aspect ratio, or distance between adjacent elements [2]. Thus, both individual elements and their interactions are being investigated due to their importance in future technological applications.

There are many methods to grow magnetic nanowires. Here, we use focused electron beam-induced deposition (FEBID), which consists of the local chemical vapour deposition of a gas adsorbed on a substrate, where molecules are decomposed by the interaction with a focused beam of electrons with energies in the keV range [3,4]. FEBID is a ‘‘direct writing’’ nanolithography technique, needing neither resists nor templates, which gives it unique advantages for the deposition of nanostructures in complex substrates.

We have previously deposited cobalt nanowires using FEBID [5,6] and their magnetic properties have been measured by magnetic force microscopy [7]. Sensors based on nanomechanical structures [8] have reached unprecedented levels of sensitivity. It has been demonstrated that mass sensors based on carbon nanotube resonators can achieve mass resolutions of the order of a single proton [9]. There is therefore a growing interest in bridging nanomechanics with nanomagnetism. For example, Rugar et al. [10] detected a single spin by magnetic resonance force microscopy, although this required low temperature and vacuum. Additionally, Losby et al. [11] performed nanomechanical magnetometry of permalloy cantilevers.

Standard scanning probe microscopy approaches use the reflection of a laser beam from the cantilever to measure its deflection. This strongly limits their application when there are geometrical constraints (e.g. between the poles of an electromagnet) and also has problems for cantilevers smaller than the laser wavelength. Self-sensing (piezoresistive) cantilevers avoid all these problems, at the expense of typically having lower resolution due to a higher electronic noise. It has been shown, however, that piezoresistive cantilevers can also be fabricated with a very high resolution, being able to resolve a mass below 1 ag in air at room temperature [12]. In fact, piezoresistive cantilevers have been considered for magnetometry for some time [13].

We have developed piezoresistive cantilevers optimized for force resolution [14-16], for applications of biomolecular recognition and biosensing [17]. In view of their force sensitivity and resolution, these cantilevers can be used for magnetometry.

In this work we present the first results on the magnetic characterization of cobalt wires grown by focused electron beam-induced deposition and studied using static piezoresistive cantilever magnetometry. In the presence of an external magnetic field, the magnetic cobalt wires deposited on the free end of the cantilever become magnetized, which leads to its magnetic field dependent static deflection.

2 Experimental procedures

The fabrication technology for the piezoresistive cantilevers has been described in [15,16]. The cantilevers are made on (100) oriented crystalline silicon on SOI wafers with a device layer thickness of 340 nm. The piezoresistor (i.e. the deflection transducer) is embedded into the crystalline silicon layer. It is defined by arsenic implantation (to minimize the junction depth) and it is oriented parallel to the cantilever axis, the <100> Si crystalline direction, in order to maximize the longitudinal piezoresistive factor. The piezoresistor is part of a symmetric Wheatstone bridge (WB) so that when it deflects, the differential output voltage of the WB changes (Fig. 1).

The cantilevers have a U-shaped structure (see Fig. 1, right), with a length of 250 μm and a thickness of about 450 nm in the legs (piezoresistors) and 325 nm elsewhere. In Table 1, the typical mechanical and electromechanical

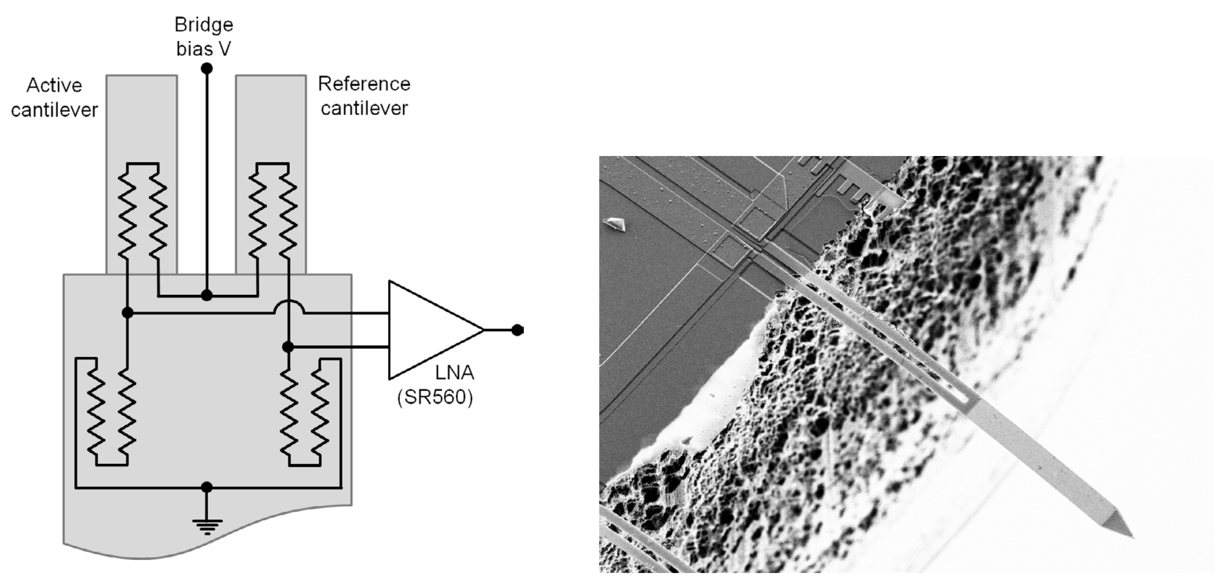

Fig. 1: Left: electrical scheme of the force sensor. The WB is composed of four n-doped silicon resistors. Two are integrated into the substrate of the chip and two into the two cantilevers, of which one acts as reference and the other is the active. Right: SEM micrograph of a piezoresistive cantilever.

Table 1: Mechanical and electromechanical characteristics of the three different piezoresistive cantilever force sensors: leg and cantilever widths, spring constant, force sensitivity, voltage noise and minimum detectable force.

Design	Leg width [μm]	Cant. width [μm]	k [mN/m]	ΔV/F [μV/nN]	V_{noise} 1Hz-1kHz [μV]	MDF [pN]
B1	2	8	0.62	358	6.2	17
B2	8	20	2.02	120	19.0	158
B3	6	16	1.73	142	3.7	26

characteristics of three different cantilever designs are reported. The electromechanical characterization of the force sensors is performed at wafer level by applying known force and displacement to the cantilever with an AFM [16]. Figure 2 shows the typical displacement sensitivity and noise power spectral density measurements for a device of the B1 design.

High quality polycrystalline cobalt microwires have been grown by FEBID [5-7] onto the free end of these three different piezoresistive cantilevers using a commercial dual beam instrument (Helios 600 NanoLab from FEI). The wires have nominal dimensions of length l=10 μm, thickness t=1 μm and width w=1 μm and are oriented parallel or perpendicular to the cantilever axis (Fig. 3A and B). Using dicobalt octacarbonyl [$Co_2(CO)_8$] as a precursor gas, high-purity wires can be grown with the following process parameters: precursor temperature=23ºC, beam voltage=5kV, beam current=11 nA (or 2.7 nA for the softest cantilever), substrate temperature=22ºC, base pressure=1x10^{-6} mbar and process pressure=6x10^{-6} mbar. From previous EDS measurements, wires grown in these conditions reach more than 90% atomic cobalt content [18].

We have used two different strategies for the fabrication of the microwires on the cantilevers. In the first approach, the deposits are done on the free end of cantilevers with no support i.e. freely suspended in vacuum (see Figure 3C). In this method, once the deposition of a magnetic microwire is done, the device is fully functional and no further processing is needed. This avoids any damage caused to the deposited structure by additional manipulation. However, we have observed that the cantilevers are bent during the cobalt deposition process, both in the working area as well as close to the support. Such deformation of the cantilevers during the deposition process leads to imperfections in the microwire shape and makes the fabrication of more than one microwire per cantilever impossible. Bending where the microwire is being grown can be explained by the stress induced by a strong local heating with focused electron beam and poor heat dissipation [19]. On the other hand, the bending close to the tip of the cantilever can be also attributed to charge accumulation in the insulating part of the device [20]. The first effect can be avoided by lowering the electron beam current but then the deposit quality can

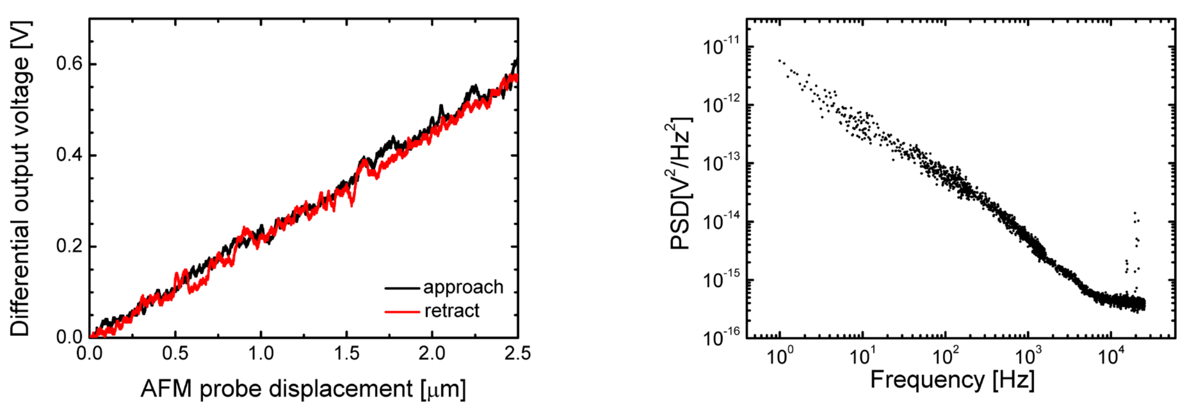

Fig. 2: Displacement sensitivity (left) and noise power spectral density (right) of the piezoresistive cantilevers (B1 design).

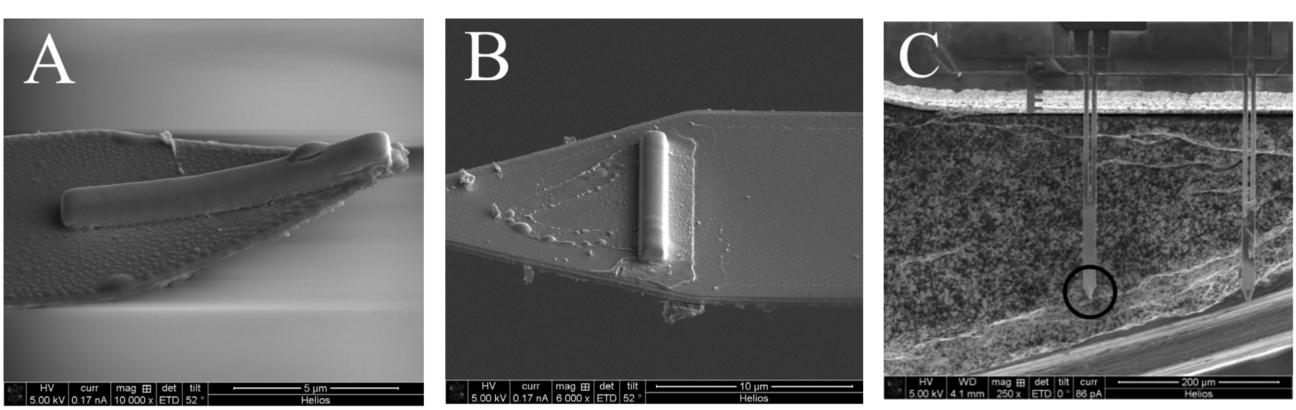

Fig. 3: SEM images of cobalt deposits on the free end of a suspended cantilever, parallel (A) and perpendicular (B) to its axis. C: view of free cantilevers in the process chamber. The free end of the cantilever is marked.

be altered [18]. The latter requires a careful grounding of the device which could not be fully accomplished in our experiments. However, it can be seen in Figure 3B, the stress induced by the wire growth is not sufficient to bend the cantilever tip if it is grown perpendicular to the long axis of the beam. In fact, in this direction, the cantilever bending stiffness is much larger so that the bending is not appreciable.

We have thus explored a second strategy, consisting of fabricating microwires on the cantilevers that were still embedded in the wafer and not yet released. A clear advantage of this approach is that the deposits are grown in optimal conditions for both shape and composition (see Figure 4). However, the drawback of this approach is the need to release the cantilevers after the microwires have been grown. This is done by a deep reactive ion etching process, which may alter the microwire composition or shape. This can be especially delicate on the edges and could produce a modification of the expected magnetization process in the wires.

For measuring the magnetic properties of the microwires, the force sensor is placed into an electromagnet, the WB is biased by 5 V DC and the magnetic field, H, is swept from -10 kOe to 10 kOe. At the same time, the variation of the differential output voltage of the WB is recorded after amplification and filtering of the signal using a commercial low noise voltage amplifier (SRS 560). The cantilever is aligned to be perpendicular to the magnetic field.

The highest deflection of the cantilever is expected to happen in the experimental configuration shown in Figure 5A. In this configuration, the torque exerted by the magnetic field will deflect the cantilever upwards (or downwards, depending on the right or left direction of the wire magnetization), trying to produce the alignment of the long side of the magnetic microwire with the magnetic field. One can estimate the force exerted on the cantilever in this configuration by equaling the magnetic and mechanical torques. For example, at 5 kOe, and taking into account the dimensions and magnetization of cobalt, the magnetic torque will be 5.75 x 10^{-13} J. Given the length of the probe (2.5 x 10^{-4} m), the force acting on the cantilever tip will be 2.3 nN. However, in our case, this optimal experimental configuration is not feasible due to space limitations and the unknown initial deflection of the cantilever. As a consequence we use another geometry which also shows the existence of this phenomenon. The magnetic field is applied forming a certain angle with

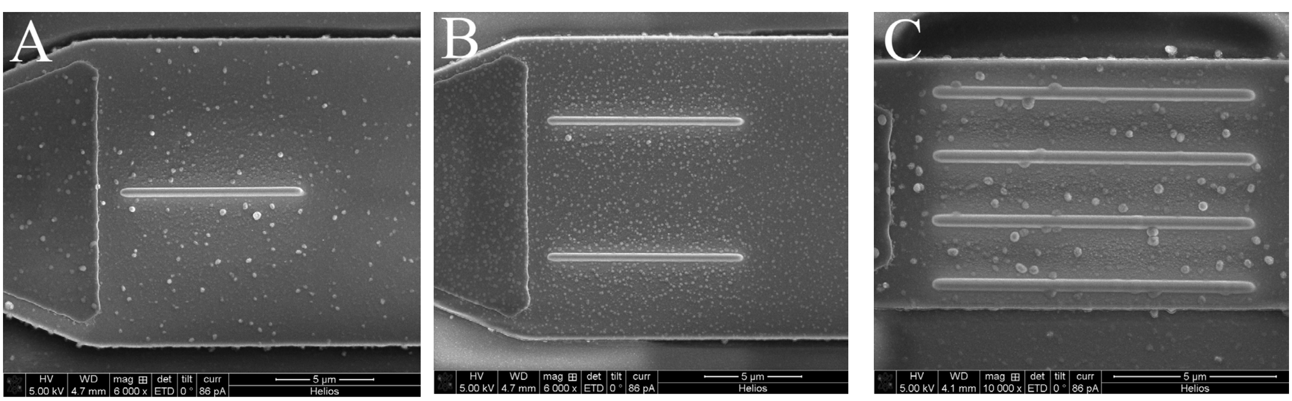

Fig. 4: SEM images of one, two and four cobalt microwires (A, B and C respectively) deposited on the cantilevers embedded in the wafer.

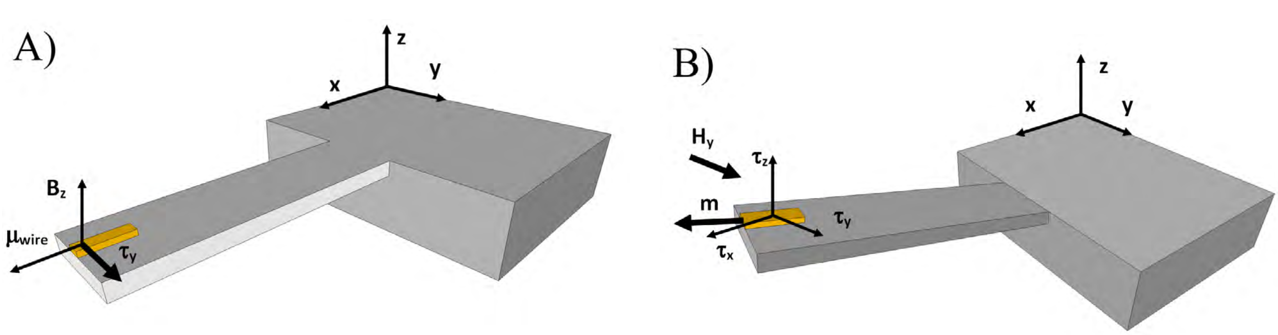

Fig. 5: A) Sketch of the optimal experimental configuration. B) Sketch of the more general experimental configuration, where the magnetic field forms an arbitrary angle with the wire magnetization.

the long direction of the magnetic microwire. We cannot know this angle precisely due to the limited control of the positioning of the cantilever with respect to the magnetic field direction as well as the unknown initial deflection of the cantilever. This more general geometry of the measurement is sketched in Figure 5B. As will be shown later, in this second geometry we are able to retrieve about half the maximum calculated signal, confirming the existence of the effect and the agreement with its quantitative estimation.

3 Results

Figures 6 and 7 show the experimental set up and a typical measurement, respectively. The results presented have been obtained with microwires fabricated using the first strategy described above. The torque (τ) exerted by the applied magnetic field on the sample magnetization is m x B, which translates into a deflection of the cantilever in the sensing direction. As shown in Figure 7, there is no cantilever deflection at zero magnetic field; the torque appears once the magnetic field increases. The wire magnetization component perpendicular to the applied field is responsible for the torque and thus for the cantilever deflection. Due to shape anisotropy, the magnetization of the wire at zero magnetic field is expected to be aligned along the long direction of the wire [5]. In Figure 7, one can notice that the torque increases for field values between 0 kOe and 5 kOe as a consequence of the increasing value of the magnetic field and thus of the m x B product. However, for higher values of the magnetic field the torque decreases due to the progressive rotation of the magnetization in the wire towards the magnetic field direction. The magnetization is fully aligned with the magnetic field for values around 10 kOe, which again gives rise to absence of torque and thus of cantilever deflection. As observed in magnetoresistance and Hall effect measurements of similar wires, the saturation field depends on the exact direction of the magnetic field with respect to the long side of the wire, but it is in the range of 10 kOe [18].

4 Conclusions and perspectives

In the present manuscript, the first successful experiment describing the use of piezoresistive cantilevers to investigate the magnetic behavior of magnetic structures grown by FEBID techniques is reported. It represents a proof-of-concept of the viability of the method, which is currently being developed for more advanced magnetic characterization of the wires. Given the good signal-to-noise ratio in the measurements displayed in Figure 7, which have been taken at room temperature without real control of the ambient conditions, there is significant margin for pushing this approach towards the detection of smaller magnetic nanostructures. Furthermore, a more refined experimental configuration would produce higher output signal in the piezoresistive cantilevers (a factor of two theoretically). As a consequence, this technique could be used to study magnetization processes in small magnetic nanostructures and in magnetic bacteria in liquid [21] when more standard techniques cannot be used.

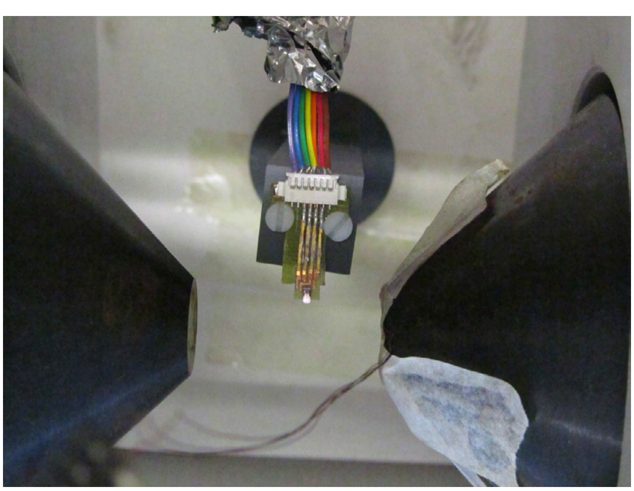

Fig. 6: Experimental setup with cantilever chip in the poles of the electromagnet.

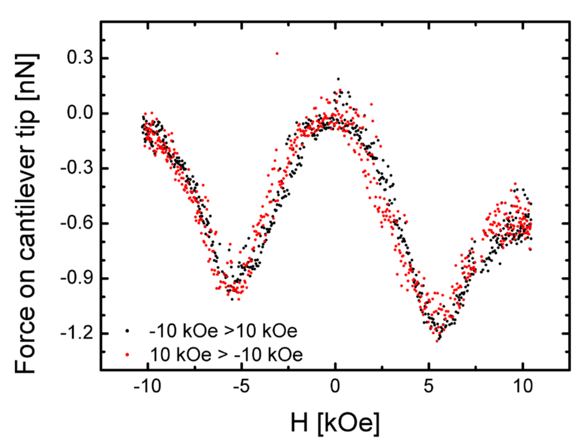

Fig. 7: Force measured by the cantilevers from the differential output voltage of the WB vs. the applied magnetic field H. The black and red color points correspond to sweep H from negative to positive values or the opposite, respectively.

The approach presented here complements other studies regarding the use of cantilevers with functionalized magnetic structures grown by FEBID. In particular, previous results have shown that this technique is suitable for creating tips for Magnetic Force Microscopy [22] or for Ferro-Magnetic Resonance Force Microscopy [23] (see also H. Lavenant et al., submitted to this Special Issue). The results presented here emphasize the flexibility of the FEBID technique, in terms of type of substrate (in this case piezoresistive cantilevers), for the growth of magnetic structures, enlarging its range of applications.

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