An individual iron nanowire-filled carbon nanotube probed by micro-Hall magnetometry

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(Received 18 October 2010; accepted 4 November 2010; published online 22 November 2010)

We report on the magnetic properties of an individual, high-quality single-crystalline iron nanowire with diameter \(d=26\) nm. The nanowire is embedded in a carbon nanotube which provides complete shielding against oxidation. Magnetization reversal is associated with domain wall formation where domain nucleation is initiated by curling. The observed nucleation fields of up to 900 mT are much higher than reported previously and nearly reach the shape anisotropy field of iron nanowires.

The properties of individual nanoscale magnets have attracted much attention over the past two decades, in part motivated by potential applications such as spintronic nanodevices, high density magnetic data storage, and biomedical applications.1–9 Iron nanostructures form an ideal test system to study the effect of size reduction on magnetic properties. In addition, iron nanowires exhibit stable magnetization vectors and well separated magnetic poles which renders them attractive for various applications, e.g., in magnetic force microscopy (MFM) or cell manipulation.10–12 However, iron’s strong tendency to oxidize is a serious drawback, both for sensor surface. The Hall coefficient \(R_H\) and magnetic field range \(B^\parallel=1.7\) T under study. Magnetic fields were applied in the sensor-plane and the angle \(\Theta\) between nanotube and magnetic field was varied by rotation of the device.

Our experimental setup probes the Hall voltage generated by the Fe-CNT’s magnetic stray field \(z\)-component \(B_z\) (perpendicular to the sensor surface) penetrating the active area of the sensor as a function of an external magnetic field. Our local measurement technique is mainly sensitive to the stray field at one end of the magnetic wire. Since MFM studies under applied magnetic fields on our nanowires (see, e.g., Ref. 13) have clearly confirmed that the remanent magnetization state is always a single domain state the data can be straightforwardly attributed to the magnetic properties of the entire wire. Data are obtained at field sweeping rates \(\nu=0.1–0.4\) T/min at various temperatures \(T\) and angles \(\Theta\) of the external magnetic field \(\mu_B\mathbf{H}\) direction with respect to the Fe-CNT long axis. Typical results of magnetization loops are shown in Fig. 2. At large angles [Fig. 2(a)], the curves are rectangular and the main features are sharp jumps of \(\langle B_z\rangle\)

FIG. 1. SEM image of the 26 nm in diameter iron nanowire inside the CNT placed at the center of the 0.8×0.8 \(\mu m^2\) Hall device. Inset: SEM back scattered image of the Fe-CNT.

system inside a scanning electron microscope (SEM) was utilized to select a single high-purity, catalyst-free, and homogeneously filled Fe-CNT. It was placed with a precision of a few tens of nanometers on the active area of the magnetometer forming an angle of \(\approx12^\circ\) with respect to the surface (Fig. 1). Magnetic fields were applied in the sensor-plane and the angle \(\Theta\) between nanotube and magnetic field was varied by rotation of the device.

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which are symmetric with respect to $H=0$. These jumps at fields $H_n$ are associated with magnetization reversal of the encapsulated nanowire. We note that this behavior resembles single domain particle magnetization reversal. At small angles $|\Theta| \leq 37^\circ$, however, the magnetization loops reproducibly exhibit additional small jumps which will be discussed below in terms of nucleation of a domain with reversed magnetization at $H_n$ followed by domain wall depinning at slightly higher fields [Fig. 2(b)].

Upon changing the angle $\Theta$ of the applied magnetic field we observe a strong change of $(H_n)$ as summarized in Fig. 3. At small angles, magnetization reversal only moderately depends on $\Theta$ and we find $\mu_0(H_n) \approx 265 \text{ mT}$. For $\Theta > 45^\circ$, however, there is a strong increase of $(H_n)$ and very high nucleation fields are observed when $\Theta \rightarrow 90^\circ$. In nearly perpendicular configuration a maximum $\mu_0(H_n)$ of 891 mT is found which is close to the theoretically predicted shape anisotropy field $(\mu_0H_A=2K_f/M_s)$ of about 1.1 T for an infinitely long iron cylinder.\(^\text{17}\) Recalling that, e.g., imperfections reduce the experimentally observed nucleation fields in any real material, this finding confirms the extraordinary high quality of the iron nanowire inside the CNT.

The angular variation of $(H_n)$ (Fig. 3) is typical for switching via the curling mode in ferromagnetic nanowires. In this model, $(H_n)(\Theta)$ for infinitely long ferromagnetic cylinders is described by\(^\text{18}\)

$$H_n = \frac{M_s}{2} \frac{a(1+a)}{\sqrt{a^2 + (1+2a)\cos^2(\Theta)}}$$ (1)

Here, $M_s$ is the saturation magnetization of iron and $a=-(d_0/d)^2$, where $d$ denotes the nanowire’s diameter and $d_0=2\sqrt{A/\mu_0M_s^2}$ is the so-called exchange length depending on the exchange constant $A$. Describing the data in terms of Eq. (1) reveals a good agreement with the experimentally observed angular dependence of $(H_n)$ (cf. line in Fig. 3). Quantitatively, fitting yields $d_0=12 \text{ nm}$ and $\mu_0M_s=2.2 \text{ T}$ which agrees well with literature data.\(^\text{19}\)

While the experimental data $(H_n)(\Theta)$ nicely agree to the curling model for an infinitely long nanowire, the observation of a second step in $M(H)$ at small angles [Fig. 2(b)] implies a more complex scenario. We recall that in real systems even for high-quality nanowires the localized magnetization reversal mechanism is often favored.\(^\text{20,21}\) Here, nucleation of a magnetic domain with reversed magnetization and a corresponding domain wall appears at the end of the nanowire. Accordingly, complete magnetic switching is associated with rapid domain wall motion through the nanowire. This scenario is supported by the observed additional magnetization steps at small angles [Fig. 2(b)].

A detailed study of the distribution of nucleation fields (inset of Fig. 3) provides further evidence for this scenario. At low temperatures the distribution of nucleation fields splits into two well separated parts while no splitting occurs at higher temperatures $T \geq 20 \text{ K}$. This splitting implies at least two reversed domain nucleation sites characterized by different energy barriers which become distinct at low temperatures.

Finally, we discuss the temperature dependence of $(H_n)$ as displayed in Fig. 4 for a fixed angle $\Theta=13^\circ$, i.e., in the two-step regime of $M(H)$. As expected for a thermally activated process, the nucleation field decreases upon heating. Quantitatively, in the thermal activation regime the mean nucleation field is described by\(^\text{22}\)
The activation volume constant, $E$, to a localization of the nucleation mode and shows that the conjecture that any arbitrarily weak inhomogeneity can lead to the reversal of domain nucleation via domain wall formation. Depending on inhomogeneities may lead to domain wall pinning followed by a domain wall propagation. Depending on $\Theta$, the pinning field may be higher than the curling instability.

$$\langle H_n \rangle = H_0 \left[ 1 - \left( \frac{k_B T}{E_0} \ln(cT/v) \right)^{2/3} \right],$$

(2)

where $H_0$ is the nucleation field at $T=0$, $k_B$ the Boltzmann constant, $E_0$ the mean energy barrier at $H=0$, $v=3.3$ mT/s the sweeping rate of the magnetic field and $c=k_B H_0/(E_0 \tau_0)$, with $\tau_0$ the prefactor of the thermal activation rate. We use $\tau_0=1.2 \times 10^{-10}$ s. Fitting the experimental data by means of Eq. (2) yields $E_0/k_B=5.18 \times 10^4$ K and $\mu_0 H_0=272$ mT. The activation volume $V=E_0/(\mu_0 M_0 H_0)$ derived from $E_0$ only amounts to $V=1.5 \times 10^3$ nm$^3$. This volume is much smaller than the one of the nanowire ($=8 \times 10^6$ nm$^3$) which again implies that the reversal starts in a small region of the wire, consistent with domain nucleation.

Although the angular dependence of $\langle H_n \rangle$ indicates the evolution of curling modes, there is compelling evidence of localized magnetization reversal via domain wall formation and propagation. The latter observation is consistent with the conjecture that any arbitrarily weak inhomogeneity can lead to a localization of the nucleation mode and shows that the actual degree of localization strongly depends on the nanowire’s structure. In contrast to other experimental work, where magnetization data deduced from magnetoresistance measurements of Ni and Co nanowires only reproduce the curling mode for angles $<50^\circ$, our data agree with the curling model for large angles, too. This is somewhat surprising, as theoretical calculations suggest a transition from curling to coherent rotation at a certain angle $\Theta$ which we do not observe in our studies. Our data hence suggest a scenario in which switching starts by the nucleation of a small domain with reversed magnetization according to the curling model where inhomogeneities may lead to domain wall pinning followed by a domain wall propagation. Depending on $\Theta$, the depinning field may be higher than the curling instability.

In conclusion, we have investigated the magnetic properties (temperature and angular dependence of the nucleation fields) of a long CNT-coated single crystalline Fe nanowire with diameter $d=26$ nm by means of micro-Hall magnetometry. The angular dependence of the nucleation field is in good agreement with the curling model for infinitely long ferromagnetic nanowires. However, the observation of additional magnetization steps at small angles, the distribution of the nucleation fields, and our analysis of the temperature dependence provide good experimental evidence that magnetization reversal is associated with reversed domain nucleation, supporting a scenario where domain formation is initiated by curling. At angles $\Theta$ close to $90^\circ$ we detect extraordinary high nucleation fields (up to $900$ mT) near the theoretical limit, implying that Fe-CNT is a very promising nanomaterial for applications where environmentally protected nanomagnets with extreme anisotropy are demanded.

Work was supported by the EC (CARBIO: MRTN-CT-2006-035616) and the DFG (Grant No. KA1694/5). We thank Nanonic GmbH Co. and J. Biberger for technical support and C. Linz for contacting the two-dimensional electron gas. Valuable discussions with P. Das and J. Müller are gratefully acknowledged.