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Hall magnetometry on a single iron nanoparticle

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High-sensitivity magnetometry over a wide temperature range has been achieved using submicron GaAs/GaAlAs Hall gradiometry. The sensitivity and versatility of the technique was demonstrated by the successful measurement of the magnetization switching of a single Fe nanoparticle with $m \sim 5 \times 10^5\ \mu_B$ ($\sim 5 \times 10^{-15}$ emu) at temperatures as high as 75 K. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1487921]

Magnetism on the nanometer scale continues to be the focus of extensive research due to its fundamental importance as well as its technological relevance in magnetic sensing and information storage. Numerous mature as well as novel nanofabrication techniques have been employed or developed to generate a wide variety of magnetic nanostructures with unprecedented density, complexity, and sophistication. In contrast, techniques for magnetic measurements down to nanometer scale are not nearly as well developed. Particularly lacking are high sensitivity and noninvasive methods that are capable of measuring individual nanoscale magnetic units over wide temperature and magnetic field range. One technique that has produced an impressive array of results on individual ferromagnetic nanowires and nanoparticles is micrometer scale superconducting quantum interference device (SQUID) magnetometry, where the nanowire or nanoparticle is placed on a microfabricated SQUID to maximize the flux through the loop.[1] However, the most effective micro-dc-SQUIDs so far are based on Nb/AlO_x/Nb Josephson junctions, whose operation is limited to low temperatures and small applied field. For practical applications in magnetic recording, field sensing, or biomagnetic detection, high sensitivity operations at higher temperature and even at room temperature are necessary.

Micro-Hall magnetometry, based primarily on two-dimensional electron systems (2DES) in semiconductor heterostructures, has emerged as an important magnetic measurement technique with the prospect of high sensitivity, small size, wide operational temperature, and virtually without any limitation in applied field. This method, which measures the Hall response of the 2DES to the small dipole field from the magnetic entities, has been widely applied for the measurements of a large variety of materials including superconductors,[2] ferromagnetic particles,[3] and patterned submicron- or nanomagnets.[4–6] Miniaturized Hall devices have been used effectively as scanning magnetic probes.[7,8] Marked improvement in magnetic moment resolution, especially at high fields, can be realized by using gradiometry in which an empty Hall cross with opposite current flow is employed to circumvent the difficulty of measuring a tiny signal on top of a much larger background.[3] The first Hall gradiometry measurement of nanoparticles was performed on arrays of iron particles (10^2–10^3 particles, 10^5–$10^6\ \mu_B$ per particle) grown on Hall crosses of a few μm linewidth.[3] Better sensitivity was achieved by matching the size of the array to that of the Hall cross.[9] Given the fact that the Hall signal ($\Delta V = I\langle B\rangle/ne$) in the ballistic regime is proportional to the average perpendicular stray field, $\langle B\rangle$, over the active area of the Hall cross,[10] one would expect that the sensitivity can be increased to the point of measuring a single nanoparticle by shrinking the size of Hall probes to submicron range.[9,11] Miniaturization of Hall probes is also important for scanning Hall probe microscopy in order to obtain high spatial resolution.[7,8]

In this letter, we report on the miniaturization of Hall gradiometers to submicron size, which yields sufficiently high sensitivity to measure an individual cylindrical iron nanoparticle with diameter $d < 10$ nm and magnetic moment $m < 10^6\ \mu_B$. Submicron-magnetic entities, investigated by micron or submicron sized Hall magnetometers typically[4–6] have at least two dimensions on the order of 100 nm ($\sim 10^7$–$10^9\ \mu_B$). The particle volume used in this work is at least two orders of magnitude smaller and, therefore, provides a more rigorous test on the ultimate resolution of the Hall probe devices. Moreover, this allows us to study true single-domain particles.

The most critical step in such an experiment is the alignment of the magnetic nanoparticle with the Hall cross. We accomplished high precision alignment of the iron nanoparticles using scanning tunneling microscopy (STM) assisted chemical vapor deposition to grow the particles.[12,13] Prior to the growth, the imaging mode of the STM was used to locate the Hall cross and align the STM tip precisely to the desired position. Thereafter, the precursor iron pentacarbonyl $Fe(CO)_5$ was introduced into the STM chamber and a bias voltage of -17 V was applied to the STM tip to start the growth. A constant tunneling current of 50 pA was main-

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tained during the growth until the desired height of particle was reached and the tip was moved to the next spot for growth. The fabricated particles have a magnetic core (body-centered-cubic-Fe) surrounded by a carbon coating,[12] which effectively prevents the sample from oxidation. The iron core diameter of the particles can be controlled and made less than 10 nm, mainly determined by the precursor pressure.[12] The particle height was chosen to be 100–120 nm in this study, so that the easy magnetization direction of the nanoparticles resulting from shape anisotropy is perpendicular to the substrate surface.

The Hall gradiometers were patterned from *n*-type GaAs/AlGaAs heterostructures by electron-beam lithography followed by wet chemical etching. A 40–50 nm thick gold gate was deposited on the top surface for two reasons: one is to provide a tunneling current path for the imaging and growth, and the other is to vary the electron density of the 2DES by gating if needed.

Figure 1(a) shows a scanning electron microscopy (SEM) image of an array of 16 iron nanoparticles with a height $h\sim 100$ nm grown on a Hall cross with a physical size of $\sim 0.7\ \mu m \times 0.7\ \mu m$. The 2DES is ~ 140 nm below the surface, and has a carrier concentration of $2.5\times 10^{11}\ cm^{-2}$ and mobility of $1.1\times 10^5\ cm^2/Vs$ at 77 K in the dark. Because of the edge depletion, the carrier concentration in this submicron channel decreases to $\sim 1.2\times 10^{11}\ cm^{-2}$ without gating as determined from the Hall effect, and we estimate that the active area of the cross is reduced to $\sim 0.5 \times 0.5\ \mu m^2$. Shown in Fig. 1(b) is the raw Hall voltage across the gradiometer with this array at different temperatures. The magnetic field was applied parallel to the easy magnetization direction of the nanoparticles. Since the large Hall signal of the 2DES from the applied background field has been canceled out by the compensation technique, the nonlinear background signal from magnetoresistance and mesoscopic effects can now be seen clearly. This nonlinear background for submicron patterns is much more pronounced than that in larger Hall devices, especially at the low temperatures. Despite this, we were able to obtain well-defined hysteresis loops at all temperatures. This is best evidenced by subtracting the up–sweep curve from the down–sweep curve. After the subtraction, we are left with the net contribution from the magnetization processes of the nanoparticles, which is plotted in Fig. 1(c). The data at $T=75$ K were taken at a faster field sweep rate to avoid the telegraph noise due to *DX*-centers. There are clear discrete steps in each of the magnetization curves, which correspond to the switching of only a few particles. This is in contrast to the observed magnetization switching process of the previously measured large arrays, where a smooth distribution of switching fields was usually seen. The observation of discrete switching of only a few particles indicates that the sensitivity of this Hall gradiometer is sufficient for measuring a single particle that is well positioned on the Hall cross. A slight decrease in the switching field with increasing temperature is also observed, which is likely due to thermally assisted switching and is consistent with previous results on large arrays.[14] The details of the temperature and field dependence of the magnetization will be discussed elsewhere.

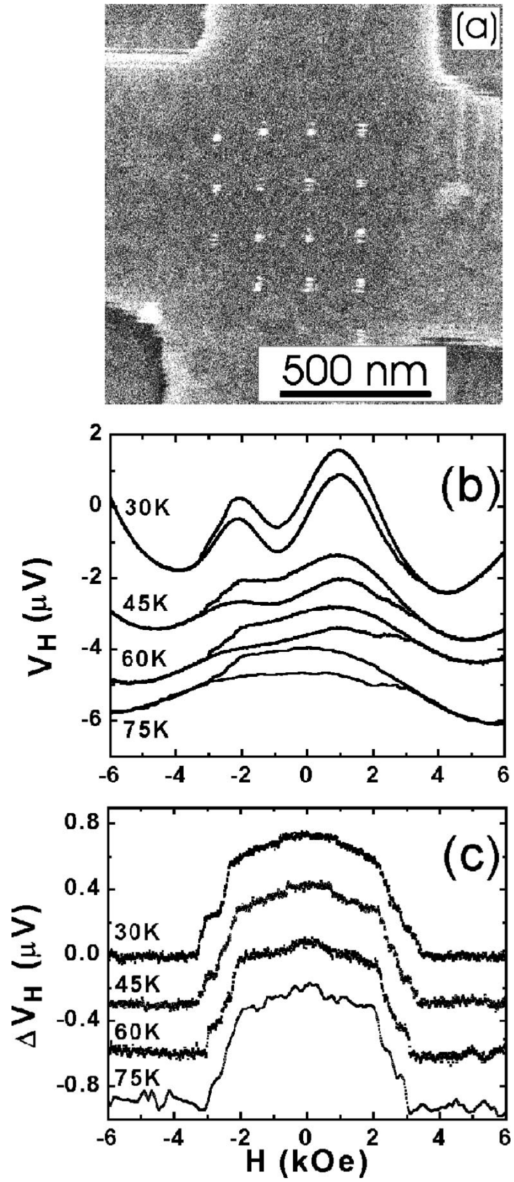

FIG. 1. (a) SEM image of an array of 16-iron nanoparticles grown on a $0.7\times 0.7\ \mu m^2$ Hall cross, (b) raw Hall voltage obtained by the compensation Hall measurements at different temperatures, and (c) Subtraction of up–sweep curves from corresponding down–sweep curves. Relative position of curves is shifted for a clearer view. In this measurement, a driving current of $\sim 0.45\ \mu A$ was used, and $R_H = 0.33\ \Omega/G$.

Figure 2 shows an SEM image of a Hall cross with a physical size of $0.6\times 0.6\ \mu m^2$ with only one iron particle (particle A) grown in the active region. This Hall gradiometer was patterned from a wafer in which the 2DES is ~ 120 nm below the surface and has a carrier concentration of $2.2\times 10^{11}\ cm^{-2}$ and mobility of $1.1\times 10^5\ cm^2/Vs$ at 77 K. A 40 nm thick gold gate was deposited on this probe. The active area of the Hall cross is estimated to be about $\sim 0.4 \times 0.4\ \mu m^2$ due to edge depletion. Simple calculation shows that the contribution from particle B to the Hall signal is about one order of magnitude smaller than that of particle A. Shown in Fig. 3(a) is the magnetization measurement of this single particle at 45 K. A single sharp switch at 2.8 kOe can be seen in the raw hysteresis curve [Fig. 3(a), inset]. By subtracting the nonlinear background from the raw Hall signal, we obtain a net hysteresis curve of nearly rectangular shape [Fig. 3(b)]. The signal-to-noise ratio obtained here is

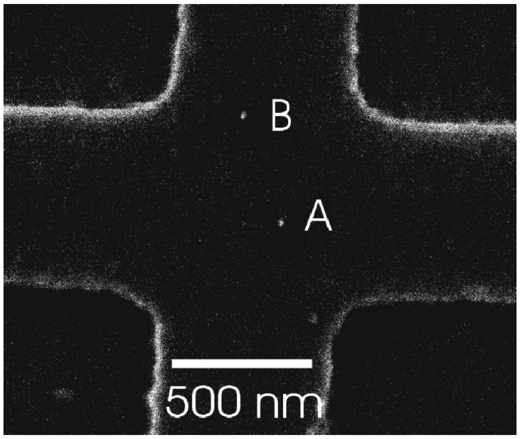

FIG. 2. SEM image of a $0.6\times0.6\ \mu m^2$ Hall cross with one particle in the active area. Particle B is about 0.35 μm from the center, and has negligible contribution to the Hall signal.

about 5, so the contribution from particle B is below the noise floor and negligible. An average stray field of $\sim$0.26 Oe due to remnant magnetization is extracted from the Hall signal. From the measured stray field, the iron core diameter of this cylinder-shaped particle ($h\sim120$ nm) is estimated to $\sim$5 nm which corresponds to a total moment of $\sim5\times10^5\ \mu_B$. Therefore, this experiment has demonstrated a moment sensitivity of $10^5\ \mu_B$ (Ref. 15) for the Hall gradiometry at temperatures and applied magnetic fields much higher than those applicable to micro-SQUIDs.

The sensitivity of the micro-Hall gradiometry still has much room for improvement. Even higher sensitivity is possible with better positioning of the particle, smaller Hall cross size, shallower 2DES, and optimized driving current.[9] For example, the aforementioned cylinder-shaped particle located precisely at the center of the same Hall cross would increase the Hall signal by $\sim$14%. Moreover, the distance between the particle and 2DES in the above sample is $\sim$160 nm. If we decrease the distance to 90 nm, the signal of this particle positioned at the same position would increase by $\sim$66%. Another factor is the noise level, which increases as the channel width shrinks due to larger mesoscopic effects. In general, miniaturization leads to significant increase in the coupling between the nanoparticles and the magnetometer, but it also decreases the field sensitivity because of the larger noise level and limitation in driving current. To optimize the performance of the Hall gradiometers, one must find the best balance point between the coupling and field sensitivity. With these optimizations we believe it is probable to realize Hall magnetometers with sensitivity comparable to that of micro-dc SQUIDs ($10^3\ \mu_B$) at room temperature and high magnetic fields.

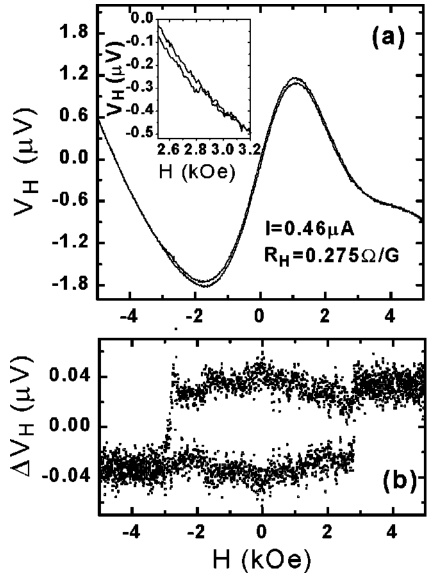

FIG. 3. Hall gradiometry measurement of a single iron nanoparticle (particle A) at $T=45$ K. (a) Hysteresis loop of raw Hall voltage with nonlinear background, Inset: a close-up view of the raw data around a magnetization switching and (b) The hysteresis loop with nonlinear background subtracted.

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