Millikelvin scanning nitrogen-vacancy magnetometry

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We report on the implementation of a scanning nitrogen-vacancy (NV) magnetometer in a dry dilution refrigerator. Using low-power, pulsed optically detected magnetic resonance combined with efficient microwave delivery through a co-planar waveguide, we reach a base temperature of 350 mK, limited by experimental heat load and thermalization of the probe. We demonstrate scanning NV magnetometry by imaging the Abrikosov vortex lattice in a 50-nm-thin aluminum microstructure. The sensitivity of our measurements is approximately 3 µT per square root Hz. Our work demonstrates the feasibility for performing non-invasive magnetic field imaging with scanning NV centers at sub-Kelvin temperatures.

Imaging magnetic fields at the micro- to nanoscale is a powerful method for materials characterization and provides insights into a plethora of physical phenomena, ranging from magnetic ordering to the flow of electric currents \cite{1}. Over the past decade, nitrogen-vacancy (NV) centers in diamond scanning probes \cite{2,3} have been introduced as versatile magnetic field sensors that are applicable over a broad temperature range. At room temperature, researchers have used NV probes to map the magnetic structure in antiferromagnets \cite{4-6} and van-der-Waals (vdW) magnets \cite{7}, and to image electronic transport in one-dimensional \cite{8} and two-dimensional conductors \cite{9-11}. At cryogenic temperatures, initial experiments focused on the imaging of vortices in iron pnictide and cuprate high-temperature superconductors with critical temperatures of $T_c = 30$ K and 90 K, respectively \cite{12,13}. More recently, vdW magnets have been studied at temperatures down to 4 K \cite{14-16}. Cryogenic scanning NV magnetometers have also been used for spatial imaging of hydrodynamic electron flow at temperatures between 5 – 100 K \cite{17,18}.

There is considerable incentive to bring scanning NV magnetometry to sub-Kelvin temperatures. Not only would this allow addressing interesting physics like superconductivity or magnetism in twisted bilayer graphene \cite{19,20} and in oxide interfaces \cite{21}, but it would also ideally complement existing scanning probe techniques such as SQUID-on-tip \cite{22-25}, scanning gate \cite{12}, and scanning tunneling microscopy \cite{26}. The biggest challenges towards millikelvin NV magnetometry are microwave-and laser-induced heating, vibrational stability of the atomic force microscope (AFM), and the experimental complexity of cryogenic instrumentation. Moreover, the low-temperature charge stability of shallow NV centers has been put into question \cite{27,28} and the phospholuminescence (PL) emission \cite{29,30} and PL contrast \cite{28,31} of NV centers have been found to decrease towards cryogenic temperatures. It is therefore an intriguing technical and scientific question of whether scanning NV magnetometry can be robustly applied in the sub-Kelvin regime.

In this work, we demonstrate scanning NV magnetometry at temperatures down to 350 mK. Experiments are carried out at the bottom of a dry dilution refrigerator and employ pulsed optically detected magnetic resonance (ODMR) detection of the NV center to mitigate microwave- and laser-induced heating. We find that NV centers are stable within the temperature range of our experiment (0.35 – 3 K) with no noticeable change in their spin and optical properties. We demonstrate scanning magnetometry by imaging the Abrikosov vortex lattice in thin aluminum micro-discs exposed to weak (0.4 – 1 mT) magnetic bias fields.

A schematic of our experimental setup is shown in Fig. 1(a-c). The setup consists of a combined atomic force/confocal microscope (AFM/CFM, Attocube) rigidly suspended from the cold insert of a CFCS110 dry dilution refrigerator (Leiden Cryogenics). The system features free-space access through a 8 mm central bore for the optical excitation of the NV spin and collection of the PL signal. The objective is fixed in space while both the sample and the diamond tip are mounted on stacks of nano-positioners and scanners for movement along all three spatial axes. The microwave (MW) drive signal needed to manipulate the NV center spin is guided to the sample through a pair of coaxial lines that are thermalized at each cooling stage of the cryostat. A superconducting vector magnet (American Magnetics Inc.) is used to apply a small out-of-plane bias field $B_z$. As a dry pulse-tube system, our refrigerator introduces noticeable vibrations between scanning tip and sample. We did not calibrate these vibrations but estimate them to be of order of tens of nanometers, similar to other rigid dry systems \cite{32}.

We glue our sample onto a copper sample holder, which also contains a resistive heater and a calibrated ruthenium oxide ($\text{RuO}_2$) thermometer ($T_{\text{RuO}_2}$), as is shown in Fig. 1(b). A cooling braid serves as a direct thermal link between the sample holder and the cold finger of the insert. The insert is in turn thermally anchored to the mixing chamber by a spring-loaded mechanical contact arm (Leiden Cryogenics). We measure a base tempera-
Figure 1. (a) Schematic of the experimental setup. The scanning NV magnetometer is located at the bottom of a dry dilution refrigerator. Optical excitation and detection use the clear central bore. Microwave pulses (MW, blue arrow) are delivered through the co-planar waveguide (CPW). Refracted laser light is indicated in green. Due to heating effects, the local temperature $T$ is indicated in green. (b) Detail of the scanning magnetometer, indicating the relative positions of scanning tip and sample. The sample holder contains both, a resistive heater and a thermometer ($T_{\text{RuO}_2}$). (c) Close-up schematic of the scanning tip over the aluminum film. The NV center is shown by a red arrow. Microwave pulses (MW, blue arrow) are delivered through the co-planar waveguide (CPW). Refracted laser light is indicated in green. Due to heating effects, the local temperature $T_{\text{sample}}$ can differ from thermometer readings ($T_{\text{RuO}_2}$). (d) Optical micrograph of the patterned aluminum film. The large structure is the CPW used for microwave delivery. The zoom-in shows the small ($1−5 \mu m$ diameter) discs placed in the gaps of the CPW that serve as test structures for scanning experiments.

Our experiment makes use of two nanofabricated structures, including the sample and the scanning tip. The sample consists of a thin aluminum film structured into a co-planar waveguide (CPW) for microwave delivery as well as several micron-sized discs for demonstrating scanning magnetometry. Aluminum is an ideal material for our demonstration because it becomes superconducting below $T_c \sim 1.25 \text{ K}$ [33], allowing us to use the Meissner effect as a magnetic hallmark. Both the CPW and discs are lithographically patterned on a sapphire substrate in a two-step process. We first define the impedance matched, tapered CPW with a thickness of 150 nm. We then add discs of 50 nm nominal thickness and various diameters ($1−5 \mu m$) inside the CPW gaps. See Fig. 1(c) for a schematic side-view of the CPW and the discs. An optical micrograph of the sample is shown in Fig. 1(d). The scanning tip is made from a monolithic block of single-crystal diamond (100 surface orientation) using a series of dry etching steps [3, 34, 35]. The diamond tip is attached via a silicon handle structure to a quartz tuning fork oscillator for distance control. NV centers are created by low energy ion implantation. We use tips from two batches in the course of this study; all tips are from QZabre AG [36].

We start experiments by recording a set of ODMR spectra as a function of stage temperature $T_{\text{RuO}_2} = 0.35 − 1.4 \text{ K}$. We use a pulsed ODMR scheme (Fig. 2(a) and Ref. 37) to both decrease the line width of the spin resonance and reduce the duty cycle of laser and microwave irradiation. Fig. 2(b) shows representative spectra from tip #2. The measurements show that the PL contrast and resonance line width are independent of temperature. To extract the transition frequency and PL contrast, we fit a double-Gaussian to account for the hyperfine-splitting caused by the the $^{15}\text{N}$ nuclear spin. The PL contrast of a single hyperfine-transition for tip #2 ($\sim 7.5\%$) is comparably low; other tips showed contrast values of up to 15%. On average, we observe a $\approx 30\%$ reduction compared to the values measured at room temperature [31]. (Note that since we are only driving one hyperfine transition, PL contrast values are roughly one-half compared to those measured by standard ODMR [37].) Furthermore, none of the ca. 10 scanning tips tested at millikelvin temperature showed unexpected bleaching or loss of contrast. Notably, tip #1, which was used for all scanning magnetometry experiments (Fig. 3 and 4), has been in continuous operation at cryogenic conditions for more than one month without showing signs of deterioration. Overall, Fig. 2 demonstrates that NV centers in scanning tips can be successfully employed down to at least 350 mK with no adverse effects.

We proceed to demonstrate scanning magnetometry on the aluminum discs above and below the superconducting...
transition temperature $T_c$ at varying out-of-plane magnetic fields $B_z$. To record a magnetic field map, we scan the tip over the sample in contact mode and acquire a pulsed ODMR spectrum at each pixel. We obtain the local stray field $\Delta B$ from the frequency shift of the resonance (determined by a least-squares fit, see Fig. 2(b)) compared to a far-away reference location, and converting the frequency shift to units of Tesla using the NV spin’s gyromagnetic ratio ($\gamma = 2\pi \times 28 \text{GHz/T}$) [35]. Note that the frequency shift is proportional to magnetic field component along the NV symmetry axis, which lies at an $\sim 55^\circ$ angle with respect to the out-of-plane direction for our tips.

Over the course of our study, we have recorded magnetic images of multiple aluminum disks of the same size, which all show the same magnetic behaviour. In Fig. 3, we present magnetometry images taken on two representative aluminum discs, together with the sample topography (Fig. 3(a)) and a schematic phase diagram (Fig. 3(b)). When performing magnetometry at 3K, which lies above $T_c \approx 1.25 \text{K}$, we record no change in the magnetic field above the disc (Fig. 3(c)). Upon cooling the sample to the fridge’s base temperature ($T_{\text{RuO}_2} = 0.35 \text{K}$) a spatially diverse magnetic pattern emerges, indicating the onset of superconductivity in the sample (Fig. 3(d)). Notably, we observe the formation of an Abrikosov vortex lattice on the aluminum disk: while bulk aluminum is a type I superconductor, which would display complete expulsion of the magnetic field (Meissner effect), thin films of aluminum have been reported to exhibit type II superconducting properties including the formation of a vortex state [38]. At $B_z = 0.4 \mu \text{T}$, two vortices are visible (Fig. 3(d)); once we raise the field to 1.0 $\mu \text{T}$, the number of vortices increases to approximately 8 (Fig. 3(e)). At the same time, the vortex cores shrink to approximately half their initial size, and the peak magnitude of the magnetic signal decreases from 140 $\mu \text{T}$ to 70 $\mu \text{T}$. Both effects – the reduction of the core size [39] and the continuous decrease in magnetic susceptibility with temperature and field [40] – correspond to the expected behavior for type II superconductors. We further observe a clustering of several of the vortices in Fig. 3(e), possibly due to pinning at defect sites such as grain boundaries [41]. As we increase $B_z$ to 6$\mu \text{T}$, which is well above the upper critical magnetic field $B_{Zc}$, superconductivity is completely suppressed and we again record a uniform magnetic signal above the disc (Fig. 3(f)).

In this final scan, the settings were chosen to yield particularly narrow hyper-fine lines, which allows us to estimate the achievable magnetic-field sensitivity. Normalizing the pixel-by-pixel variation to a one-second integration time, we obtain a sensitivity of about $14 \mu \text{T}/\sqrt{\text{Hz}}$. This number is likely affected by the already sizeable off-axis field. Evaluating the line scans discussed further below (Fig. 4(a)), which were taken with comparable frequency resolution but at a smaller 1$\mu \text{T}$ field, yields a considerably improved sensitivity of $3 \mu \text{T}/\sqrt{\text{Hz}}$.

We finally investigate the influence of microwave and laser pulses on the sample temperature. Since the RuO$_2$ thermometer is located a few mm away from the sample (see Fig. 1(c)), the true sample temperature $T_{\text{sample}}$ is likely higher than the measured temperature $T_{\text{RuO}_2}$. To characterize the local heating, we perform NV magnetometry line scans across the edge of the aluminum disc (dashed line in Fig. 3(e)) and observe the peak field $\Delta B_{\text{max}}$ near the sample edge. Because $\Delta B_{\text{max}}$ is approximately proportional to temperature near $T_c$ [40], we can use the peak field as an indicator for the sample temperature $T_{\text{sample}}$.

Fig. 4(a) presents line scans as a function of stage temperature $T_{\text{RuO}_2}$. We regulate the stage temperature via the built-in sample heater (Fig. 1(c)) and a PID controller. We investigate three laser and microwave power...
settings, from low to high: (i) $P_{\text{laser}} = 50 \, \mu W$ and $P_{\text{mw}} = 70 \, \mu W$, (ii) $P_{\text{laser}} = 50 \, \mu W$ and $P_{\text{mw}} = 300 \, \mu W$, and (iii) $P_{\text{laser}} = 140 \, \mu W$ and $P_{\text{mw}} = 300 \, \mu W$. The given powers are time-averaged over the respective measurement sequence. Laser powers are measured at room temperature in front of the objective. Microwave powers reflect the nominal input power minus 5 dB to account for the attenuation of the cold coaxis lines.

In Fig. 4(b), we plot the peak field $\Delta B_{\text{max}}$ as a function of $T_{\text{RuO}_2}$ for all datasets (i)-(iii). By applying linear fits (solid lines), we extrapolate the critical stage temperature $T_{\text{RuO}_2}^{(c)}$ above which superconductivity vanishes. We use a common slope parameter when fitting the three datasets, and we exclude points that do not carry signal, i.e., points that lie above $T_{\text{RuO}_2}^{(c)}$. The so-determined critical stage temperature $T_{\text{RuO}_2}^{(c)} \leq T_c$ is in general lower than the critical temperature of the superconductor $T_c$ due to the temperature difference between RuO$_2$ thermometer and sample. For the lowest power setting (i), however, we observe that the extrapolated $T_{\text{RuO}_2}^{(c)} \approx 1.27 \, K$ is very close to the $T_c = 1.25 \, K$ expected for 50-nm-thick aluminum films, for which $T_c$ approaches bulk values [33]. Therefore, for (i), the local heating by microwave and laser pulses is negligible.

To corroborate these results and estimate the actual $T_c$ of our sample, we perform complementary electrical transport measurements on a separate aluminum film of similar thickness ($\sim 60 \, \text{nm}$). Fig. 4(c) shows the differential electrical resistance $dU/dI$ as a function of applied current $I$, at $T_{\text{RuO}_2} = 1.07 - 1.27 \, K$. The sharp jumps in $dU/dI$ (triangles) indicate the critical current $I_c$ below
Figure 4. (a) Magnetic line scans taken along the dashed line in Fig. 3(e). Colors indicate stage temperature $T_{\text{RuO}_2}$. Datasets (i)-(iii) are recorded using different settings with increasing microwave and laser power, as indicated by the sequence drawings (see main text for values). All data are recorded in an 1 mT out-of-plane bias field. (b) Peak signal $\Delta B_{\text{max}}$ plotted as a function of $T_{\text{RuO}_2}$ for all datasets, with error bars representing the pixel-to-pixel variations of the signal-less, high temperature scans. Solid lines are linear fits (hollow data points are excluded from the fit). A common slope parameter is used for all fits. The zero crossings (black crosses) reflect the critical stage temperatures $T_{\text{c}}$ at which the superconductivity vanishes, indicating that the sample temperature $T_{\text{sample}} = T_{\text{c}}$ at this stage temperature. The $T_{\text{c}}^{(c)}$ determined from the fit are $\approx 1.27$ K (i), 1.05 K (ii) and 0.79 K (iii), where (i) is in excellent agreement with transport measurements (dashed line, details in (c)). (c) Electric transport measurements recorded on a second microstrip similar to the one shown in Fig. 1(d). Shown is a three-point measurement of the differential resistance $dU/dI$ plotted as a function of the applied current $I$, for different stage temperatures $T_{\text{RuO}_2} = 1.07 - 1.27$ K. The sharp drops in $dU/dI$ (triangles) indicate the critical current. A finite contact resistance ($\sim 18.4 \Omega$) is subtracted from all curves.

which superconductivity sets in. With increasing temperature, the critical current is reduced and the $dU/dI$ curve becomes flatter, reflecting that the film’s temperature is approaching $T_c$. Any signs of superconductivity vanish at $T_{\text{RuO}_2} = 1.27$ K, in excellent agreement with the literature and magnetometry values.

In summary, our work extends scanning NV magnetometry to sub-Kelvin temperatures. Although our base temperature ($\sim 350$ mK) and sensitivity ($\sim 3 \mu$T/$\sqrt{\text{Hz}}$) are above those reached by state-of-the-art scanning SQUID microscopes [32, 42], there is considerable leeway for improving both figures of merit. Base temperature can be lowered by improved probe thermalization, more efficient microwave delivery [43], and possibly using low power, resonant optical readout [44]. The sensitivity can be improved using ac magnetometry [11, 18] and gradiometry [45] detection where demonstrated noise levels are below $100 \mu$T/$\sqrt{\text{Hz}}$. A remaining technical issue is spurious mechanical vibrations from the cooling circuit of the dry dilution refrigerator. With these challenges addressed, scanning NV magnetometry shows exciting promise for the non-invasive imaging of nanoscale transport, persistent ring, edge and vortex currents [42, 46], and superconductivity in the millikelvin regime [19].

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