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RPPR Final Report
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Major Goals: Diamond is a material of increasing technological and scientific importance. Compared to Si and other wide-bandgap materials, diamond has significantly higher thermal conductivity and electric breakdown field. It also outperforms Si and other wide-bandgap materials in other figures of merit relevant for electronic applications such as switching speed and power handling capacity. The importance of diamond is further enhanced by its ability to host nitrogen-vacancy (NV) centers which exhibit a spin-dependent photoluminescence. This provides a very sensitive optical means of detecting the electron spin resonance of the electron spin moment associated with the center magnetic resonance. This provides a platform for ultrasensitive spatially resolved magnetometry and for quantum technological applications. Diamond, in addition to being a stable fluorophore is also bio-compatible making it attractive for biophysical and biomedical applications. All these characteristics make diamond an important subject for material characterization studies for electronic and spintronic science and technology, and for spin based sensing applications.

We seek to characterize the properties of diamond using magnetic resonance force microscopy, or MRFM which enables spatially resolved magnetic resonance studies of wide-bandgap electronic and spintronic materials in general and of diamond in particular. We focus here on studies of the dynamics of the spins present on point defects in diamond. MRFM is a particularly powerful probe given its applicability to diverse spin species in diamond regardless of their optical activity. In particular this approach allows studies of the P1 defect center, a spin-1/2 defect that is optically inactive. Our MRFM has a spin sensitivity at the level of few electron spins enabling ESR investigation of few or single point defects.

MRFM is currently limited to force detection of magnetic resonance at frequencies much less than the resonant frequency. The recent demonstration of mechanical resonators with characteristic frequencies in the MHz regime opens the possibility of matching the detector frequency to the Larmor frequency of the nuclear spin. This approach to mechanical detection is analogous to conventional inductively detected NMR and would benefit from applying techniques developed there. Larmor detection of nuclear magnetic resonance offers important advantages over longitudinal detection. These include a) high bandwidth detection naturally reveals details of the nuclear spin dynamics; the high gradient nanomagnetic probe tip, whose position relative to the sample oscillates with the cantilever, naturally generates the large oscillating magnetic field needed to excite the magnetic resonance signal thus eliminating the need for external rf generator, c) it could enable application of powerful magnetic resonance techniques such as Fourier transform spectral analysis for spectroscopy and imaging. We seek to pursue a novel approach to NMR measurements that relies on force detection of the precessing magnetic moment directly, that is at its precession frequency, by means of mechanical detector having a resonance frequency matched to the NMR

Larmor frequency.

Accomplishments: The goals of the program are sensitive and spatially-resolved detection of magnetic resonance to enable single bio-molecule imaging. We highlight accomplishments under this funding:
1. Performed a few electron spin MRFM study of spin dynamical and transport properties in magnetic field gradient-defined, nanoscale volumes of diamond.
2. Discovered a NV-diamond based, non-resonant, broadband method of detecting the magnetic resonance of a target spin system that we label NV-NRB.
3. Used NV diamond to perform an optically detected magnetic resonance study of the structural dynamics of an individual DNA molecule.
4. High quality factors and the displacement detection sensitivity needed for NMR de-tection of nuclear spin ensembles at their nuclear Larmor frequency were demonstrated in experiments aimed toward using high frequency mechanical membrane as mechanical magnetic resonance detectors.

Our work was presented in seven publications acknowledging this source of funding. Below we discuss the advances in this program demonstrated in these four highlights. The first highlights a fundamental advance in understanding the spin properties of diamond, a material of increasing technological and scientific importance. We studied the spin proper-ties of diamond to provide foundational understanding for ultrasensitive magnetic resonance detection.

The second highlight—our discovery of a new approach to using nitrogen-vacancy (NV) centers in diamond for detection of ferromagnetic resonance—motivated further studies that advanced understanding of the use of ferromagnetic dynamics to enable sensitive NV-based optical detection of ferromagnetic resonance.

The third highlights the application of sensitive, NV diamond-based detection to biomolecular magnetic resonance. This enabled a magnetic resonance experiment on a single DNA molecule.

Fourth, we demonstrated a novel approach to mechanical detection of magnetic resonance that could expand the capabilities of mechanically detected magnetic resonance to enable direct measurement of the precessing nuclear spin magnetization at its Larmor precession frequency.

A key requirement for nanoscale magnetic resonance imaging experiments is the development of scanned probe technologies. We integrated software feedback control with scanning hardware to build sophisticated and sensitive scanned probe microscope that demonstrated simultaneous measurement of magnetic and spin transport properties of magnetic systems.

Training Opportunities: The scientific research reported here engaged undergraduate, graduate and postdoctoral scientists in the research enterprise. This engages them in the complex, multi-dimensional process of scientific exploring cutting edge research problems, using state-of-the-art research tools, engaging with diverse researchers and colleagues to become proficient in new topical areas of scientific research, collaborating to achieve results beyond the capabilities of an individual and learning to communicate their methods and accomplishments.

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as of 02-Nov-2018

Results Dissemination: This research was published in seven publications:
1. J. Cardellino, N. Scozzaro, M. Herman, A.J. Berger, C. Zhang, K.C. Fong, C. Jayaprakash, D.V. Pelekhov, and P.C. Hammel, "The effect of spin transport on spin lifetime in nanoscale systems." Nature Nanotechnology, 9(5): p. 343-347 (2014)
2. C.S. Wolfe, V.P. Bhallamudi, H.L. Wang, C.H. Du, S. Manuilov, R.M. Teeling-Smith, A.J. Berger, R. Adur, F.Y. Yang, and P.C. Hammel, "Off-resonant manipulation of spins in diamond via precessing magnetization of a proximal ferromagnet." Physical Review B, 89(18): p. 180406 (2014)
3. A.J. Berger, M.R. Page, J. Jacob, J.R. Young, J. Lewis, L. Wenzel, V.P. Bhallamudi, E. Johnston-Halperin, D. V. Pelekhov, and P.C. Hammel, "A versatile LabVIEW and field-programmable gate array-based scanning probe microscope for in operando electronic device characterization." Review of Scientific Instruments, 85(12): p. 123702 (2014)
4. R. Adur, C.H. Du, J. Cardellino, N. Scozzaro, C.S. Wolfe, H.L. Wang, M. Herman, V.P. Bhallamudi, D.V. Pelekhov, F.Y. Yang, and P.C. Hammel, "Microscopic studies of nonlocal spin dynamics and spin transport." Journal of Applied Physics, 117(17): p. 172604 (2015)
5. S.A. Manuilov, C.H. Du, R. Adur, H.L. Wang, V.P. Bhallamudi, F.Y. Yang, and P.C. Hammel, "Spin pumping from spinwaves in thin film YIG." Applied Physics Letters, 107(4): p. 042405 (2015)
6. A.J. Berger, M.R. Page, H. Wen, K.M. McCreary, V.P. Bhallamudi, R.K. Kawakami, and P.C. Hammel, "Correlating spin transport and electrode magnetization in a graphene spin valve: Simultaneous magnetic microscopy and non-local measurements." Applied Physics Letters, 107(14): p. 142406 (2015)
7. R.M. Teeling-Smith, Y.W. Jung, N.J. Scozzaro, J. Cardellino, I. Rampersaud, J.A. North, M. Simon, V.P. Bhallamudi, A. Rampersaud, E. Johnston-Halperin, M.G. Poirier, and P.C. Hammel, Electron Paramagnetic Resonance from a Single Biomolecule. Biophysical Journal, 108(2): p. 337A (2015)

These results were presented in eleven invited talks:
1. “Spin Transport via Collective Spin Excitations,” presented at the March Meeting of the American Physical Society, Baltimore MD, March 2016
2. “Spin Transport Driven by Magnetization Dynamics: Nanoscale studies of spin dynamics,” invited talk presented at the 8th ASRC International Workshop on Spin Mechanics, Tokai, Japan, 24–26 February 2013
3. “Spin dynamics and transport in nanoscale volumes,”Keynote talk presented at the 2014 Annual Meeting of the AAAS, 13–17 February 2014, Chicago, IL
4. “Probing the Influence of Interfaces in Spin Pumping,” SPIE Spintronics VII, 17–21 August 2014, San Diego, California
5. “The Role of Interfaces in Dynamic Spin Transport,” 59th Annual Magnetism and Magnetic Materials Conference, 3–7 November 2014, Honolulu, Hawaii
6. “Spin Transport by Collective Magnetization Excitations,” Santorini IV Workshop on Complex Oxides, Porquerolles, France, 16 June 2016
7. “Spin dynamics and transport in nanoscale volumes,” Condensed Matter Physics Seminar, UCLA Department of Physics & Astronomy presented 17 January 2014, Los Angeles, CA
8. “Spin dynamics and transport in nanoscale volumes,” Materials Colloquium, Los Alamos National Laboratory, 22 January, 2014, Los Alamos, NM
9. “Probing the Influence of Interfaces in Spin Pumping,” Condensed Matter Seminar, Service de Physique de l’ Etat Condense, CEA, Saclay, France, 23 September 2015
10. “Probing the Influence of Interfaces in Spin Pumping,” Seminar, SPINTEC, CEA Grenoble, France, 15 October 2015
11. “Probing the Influence of Interfaces in Spin Pumping,” Seminar, Institute for Metallic Materials (IMW) Leibniz Institute for Solid State and Materials Research Dresden, 21 October 2015

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PARTICIPANTS:

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Participant: P. Chris C Hammel 2476928
Person Months Worked: 3.00 **Funding Support:**

Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
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International Travel:
National Academy Member: N
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Project Contribution:
International Collaboration:
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National Academy Member: N
Other Collaborators:

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Project Contribution:
International Collaboration:
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National Academy Member: N
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Project Contribution:
International Collaboration:
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Other Collaborators:

Participant Type: Undergraduate Student
Participant: William Ruchotzke
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RPPR Final Report
as of 02-Nov-2018

Final Report ARO contract W911NF-12-1-0587: Nanoscale resolution magnetic resonance studies of spin dynamics and defect properties in diamond nanostructures

P.C. Hammel, Department of Physics, Ohio State University

Here we describe recent research activities supported by ARO funding under contract W911NF1210587 for the period Sep, 26 2012 to Jan, 25 2016. The broad goals of the program are sensitive and spatially-resolved detection of magnetic resonance to enable single bio-molecule imaging. We highlight accomplishments under this funding:

1. Performed a few electron spin MRFM study of spin dynamical and transport properties in magnetic field gradient-defined, nanoscale volumes of diamond [1]
2. Discovered a NV-diamond based, non-resonant, broadband method of detecting the magnetic resonance of a target spin system that we label NV-NRB [2]
3. Used NV diamond to perform an optically detected magnetic resonance study of the structural dynamics of an individual DNA molecule [3]
4. High quality factors and the displacement detection sensitivity needed for NMR detection of nuclear spin ensembles at their nuclear Larmor frequency were demonstrated in experiments aimed toward using high frequency mechanical membrane as mechanical magnetic resonance detectors.

Our work was presented in seven publications [1-7] acknowledging this source of funding. Below we discuss our advances in this program as demonstrated in these four highlights. The first [1] highlights a fundamental advance in understanding the spin properties of diamond, a material of increasing technological and scientific importance. Our study of the spin properties of diamond provides foundational understanding for ultrasensitive magnetic resonance detection.

The second highlight—our discovery of a new approach to using nitrogen-vacancy (NV) centers in diamond for detection of ferromagnetic resonance [2]—motivated further studies [4, 5] that advanced understanding of the use of

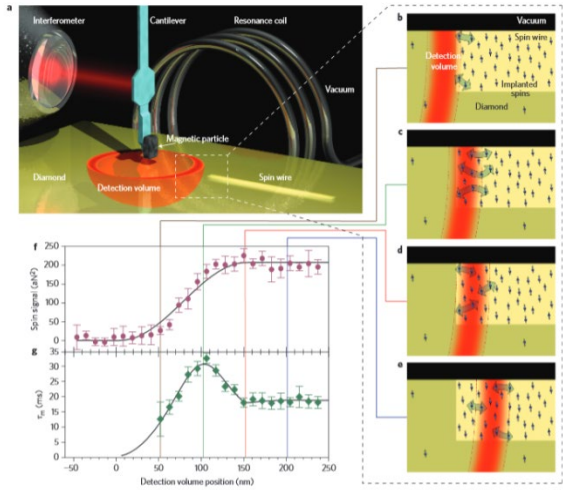

Figure 1: *MRFM spin wire setup, schematic of spin dynamics in wire, and spin noise measurements. a, MRFM setup for measuring spin noise in the 200 nm x 250 nm x 4 μm spin wire of P1 centers in diamond. As the cantilever oscillates, the slice sweeps out the spin detection volume. b-e, Schematic showing the detection volume as it is walked into the spin wire, the rectangular stripe region. The thick double arrows represent flip-flop-mediated spin diffusion in and out of the detection volume. f, The spin signal (purple circles) increases as a function of walk-in distance until the volume is fully inside the spin wire as in (e). g, The correlation time of the measured spins (green diamonds).As spins are forced to diffuse a longer distance to exit the volume, the correlation time increases, but once the measurement volume is fully inside the stripe (d and e), spins can diffuse out both sides, decreasing the correlation time to roughly half the peak value. Solid black lines show simulation results.*

ferromagnetic dynamics to enable sensitive NV-based optical detection of ferromagnetic resonance.

The third [3] highlights the application of sensitive, NV diamond-based detection to biomolecular magnetic resonance. This enabled a magnetic resonance experiment on a single DNA molecule [3].

Fourth, we demonstrated a novel approach to mechanical detection of magnetic resonance that could expand the capabilities of mechanically detected magnetic resonance to enable direct measurement of the precessing nuclear spin magnetization at its Larmor precession frequency.

A key requirement for nanoscale magnetic

resonance imaging experiments is the development of scanned probe technologies. We integrated software feedback control with scanning hardware [6] to build sophisticated and sensitive scanned probe microscope that demonstrated simultaneous measurement of magnetic and spin transport properties of magnetic systems [7].

These advances, described in seven ARO sponsored publications have advanced the prospects for ultrasensitive magnetic resonance detection applied to single molecule nuclear magnetic resonance imaging.

Equilibrium spin transport and dynamics in a diamond spin nanowire [1]

We have used nanoscale EPR [1] to probe the spins within a nanoscale channel of implanted nitrogen (P1) centers in diamond to investigate the relationship between spin lifetime and transport dynamics in small electron spin ensembles within this nanoscale spin wire [1]. Using magnetic resonance force microscopy (MRFM) [8, 9] to measure the statistical fluctuations of the ensemble magnetization [10], we observe that spin lifetime is dominated by spin transport rather than conventional spin-lattice relaxation mechanisms.

We scanned the detection volume defined by the magnetic field gradient of the tip into the spin wire **(Figure 1 (a–e))** and measured the force exerted on the cantilever by the selected spins. The measured correlation or spin memory loss time t_m, shows complex behavior arising from spin transport as the detection volume moves into the wire: ensemble correlation is lost as a consequence of the diffusion of spin into or out of the detection volume **(Figure 1).** Depending on parameter values, similar lifetime effects will impact optically detected nanoscale magnetic resonance and imaging.

Discovery of NV-NRB: Novel detection of target spins [2]

Conventional ODMR detection is based on observing changes in the optical fluorescence of an NV center in response to magnetic resonance induced transitions of its spin state [11]. We demonstrated [2] a qualitatively different method of optically detecting magnetic resonance. We find that exciting the target spins directly alters the magnetization of the NV center thus altering the NV fluroscence. We have demonstrated this for both ferromagnetic and paramagnetic target spins. The microscopic details of the underlying mechanism are under investigation.

Figure 2 shows the fluorescence intensity of NV centers in nanodiamonds dispersed on the surface of 20 nm thick YIG ferromagnetic film recorded as a function of magnetic field and microwave frequency. In addition to the standard NV ODMR powder spectrum the fluorescence is suppressed along a curve precisely following the dispersion relationship of the uniform Ferromagnetic Resonance (FMR) mode in the YIG film as shown by the solid blue line. These data demonstrate that the NV fluorescence is altered by the FMR excitation at frequencies *nonresonant* with

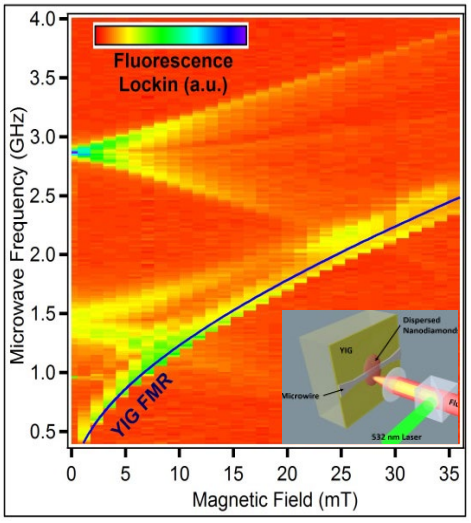

Figure 2: NV-NRB detection of proximal FM and paramagnetic centers*: Inset: Schematic of experimental setup including laser excitation of nanodiamonds (red mound), MW delivery by a patterned stripline, and fluorescence collection. NV non-resonant detection of FMR in YIG. The intrinsic NV resonance branches of the ground state and excited state begin at 2.87 GHz and 1.5 GHz, respectively, in zero-field. NV fluorescence is suppressed along the YIG FMR dispersion (labeled) far from the NV resonance conditions and over a broad range of frequencies.*

the NV center transitions themselves. The intensity of the signal is comparable to that achieved by direct microwave excitation resonant with the NV center.

The ability to optically detect magnetic resonance at arbitrary off-resonant frequencies provides a flexible and complementary approach to conventional ODMR, and hence a distinct and flexible approach to nano MRI of materials.

ESR Measurement of a Single Labeled DNA molecule using NV ODMR [3]

The dynamics of DNA molecules, important determinants of protein expression, are hard to measure and not well understood. Single-molecule EPR could provide a powerful tool for studying this. We site-selectively labeled individual double-stranded DNA molecules with a single nanodiamond containing NV centers. We detected the motion of the biomolecule and associated nanodiamond through resulting variations in the spin resonance frequency of the NV centers [3] which can be measured optically through their spin-dependent fluorescence [12].

Figure 3 schematically depicts the custom-built ODMR microscope used for the experiment. The key features are a confocal microscope, a coplanar MW antenna integrated with fluid circuits and an external magnetic field applied by a set of rare-earth magnets.

The magnetic resonance of the NV centers was detected using what we will refer to as conventional continuous wave (CW) ODMR, which is obtained by measuring the changes in fluorescence of NV centers while the microwave excitation frequency is swept. **Figure 3(b)** shows the optically detected magnetic resonance (ODMR) spectra of NV centers detected at room temperature in a single nanodiamond crystal attached to a single DNA molecule at three applied fields. The close similarity of the spectra associated with the moving nanodiamond on the end of a DNA strand to a powder spectrum confirms our expectation that the nanodiamond probe rotates isotropically, and hence freely, through all possible orientations on a timescale that is slow

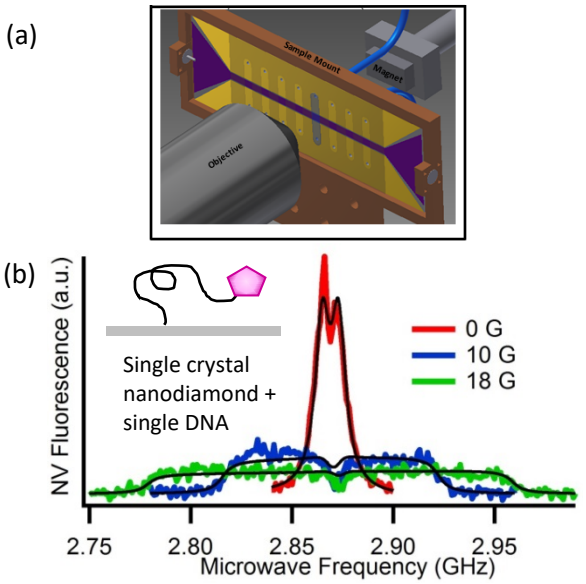

Figure 3*: a) A schematic of the custom-built confocal microscope with integrated flow cell and microwave stripline, illustrating eight flow channels for in-vitro single molecule experiment, running perpendicular across the microwave coplanar waveguide. The microwave circuit path is highlighted in purple and the fluid circuit is in blue. (b) Temporally averaged ODMR spectra of NV centers for a single nanodiamond on a DNA molecule that is twisting randomly in the liquid flow in the flow cell.*

compared to the transverse spin relaxation time T_2 for NV centers.

Toward direct detection of nuclear magnetic resonance using a high frequency mechanical membrane

Experiments on high frequency mechanical membranes that will be used for NMR detection of nuclear spin ensembles at their nuclear Larmor frequency have demonstrated the needed high quality factors and the required displacement detection sensitivity. Here, the nuclear spin precession frequency is matched to the mechanical resonant frequency of the membrane. The match in frequencies allows the radio frequency magnetic field to be produced by the motion of the detector relative to a micromagnet offering an effective and sensitive route to Larmor frequency magnetic resonance detection.

We assembled an apparatus for direct detection of nuclear magnetic resonance using a

high frequency mechanical membrane and characterized its performance in this role. In a conventional MRFM experiment, the spin magnetization is coupled to a low frequency mechanical cantilever whose mechanical resonant frequency ω_c is much lower than the Larmor frequency of spin precession ω_L. Due to the mismatch between these frequencies, external rf radiation H_1 is needed for spin manipulation, and the cantilever detects only the static component of spin magnetization. In this new approach with a high frequency resonator, $\omega_c = \omega_L$. As a result the mechanical membrane is coupled directly to the precessing components of spin magnetization via the magnetic dipolar interaction between a probe magnet and the spins under study.

The practical implementation of this approach is based on a commercial silicon nitride membrane resonators with a fundamental mechanical frequency over 1 MHz. As a result such a membrane can potentially be used as sensitive force detectors in the place of cantilevers for magnetic resonance studies. The high mechanical resonant frequency of a membrane can be matched to the Larmor frequency ω_L of a nuclear spin sample placed on the membrane. Due to experimental considerations, the sample spins are placed on the membrane and the high gradient probe magnet is placed nearby. With a high gradient probe magnet placed near the vibrating membrane, the relative motion of a sample on the membrane to the magnet can generate an rf field at the Larmor frequency of the sample, eliminating the need for an external rf generator to excite the nuclear spins which could reduce the size and cost of NMR systems.

Using a piezo actuator, we drive the membrane and observe the mechanical oscillation using optical laser interferometry. Using lock-in detection, we see the fundamental resonance frequency and higher order modes of the membrane. In our geometry, we place a ~300 micron $SmCo_5$ particle a few hundred microns above the surface of the membrane. The particle is glued onto a screw which allows us to approach the membrane while monitoring the separation with an optical microscope. Before placing the $SmCo_5$ particle in the probe, we saturated the moment of the particle using a 14 T magnetic field along the axis of the screw. This geometry makes a strong magnetic field gradient perpendicular to the membrane surface. As the membrane oscillates, spins move along the field gradient, resulting in an oscillating magnetic field that acts as the H_1 field.

Our preliminary measurement sought to observe a signal from the natively occurring Si^{29}, N^{14}, and N^{15} nuclear spins contained in the Si_3N_4 membrane. The challenges with this measurement are that Si^{29} is only about 4% abundant, N^{14} is spin 1 and has significant quadrupolar broadening which diminishes the signal, and N^{15} is less than 1% abundant. The observable signal will consist of a diminution in the quality factor of the membrane as the nuclear spins come on resonance resulting from the absorption of energy from the particle's field by the nuclear spins. Magnetic field is swept thus changing the Larmor frequency of the spins, determined by their respective gyromagnetic ratios. With the mechanical resonance of the membrane at 1.3 MHz, we

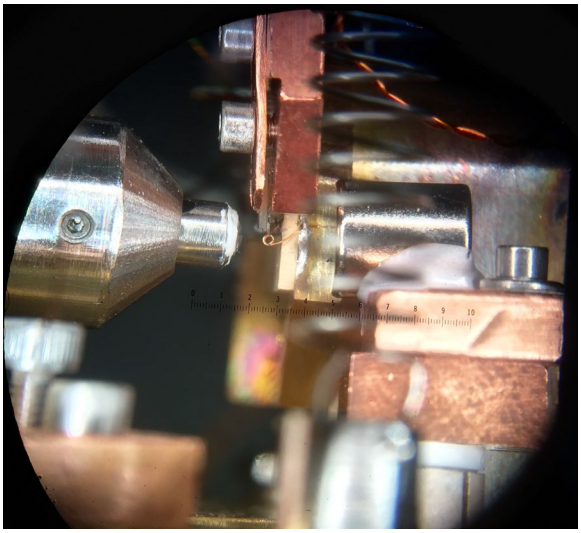

Figure 4: Apparatus for detection of magnetic resonance using a mechanical membrane. Optical fiber holder (left), permanent magnet (right) and membrane (center) are coaxially arranged.

expect resonance to occur for the Si^{29}, N^{14}, and N^{15} spins at applied fields of 1500 G, 4200 G, and 3000 G, respectively.

At the time of this report we measured the quality factor of the 30 nm thick, 0.25 mm wide membrane to be about 4 x 10^6 at 4 K. While we do see some changes in the quality factor around 2500 G, we do not see features at the expected resonance frequencies.

Ultrasoft Cantilever Magnetometry

We have developed highly sensitive cantilever magnetometry for characterization of ultrasoft cantilevers and the magnetic particles that we attach to the cantilevers. This provides an estimate of the sensitivity of the cantilever through resonance frequency and quality factor. It also measures the magnetic moment and coercivity of the tip providing important characterization information that is needed when applying these cantilevers for sensitive magnetic resonance.

We have used the same technique to measure the magnetic susceptibility of few layer graphene sheets deposited on the cantilever. These experiments could reveal important characteristics of the carriers in graphene.

Papers (7) Supported by this ARO Grant

1. J. Cardellino, N. Scozzaro, M. Herman, A.J. Berger, C. Zhang, K.C. Fong, C. Jayaprakash, D.V. Pelekhov, and P.C. Hammel, *The effect of spin transport on spin lifetime in nanoscale systems.* Nature Nanotechnology, **9**(5): p. 343-347 (2014)
2. C.S. Wolfe, V.P. Bhallamudi, H.L. Wang, C.H. Du, S. Manuilov, R.M. Teeling-Smith, A.J. Berger, R. Adur, F.Y. Yang, and P.C. Hammel, *Off-resonant manipulation of spins in diamond via precessing magnetization of a proximal ferromagnet.* Physical Review B, **89**(18): p. 180406 (2014)
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4. R. Adur, C.H. Du, J. Cardellino, N. Scozzaro, C.S. Wolfe, H.L. Wang, M. Herman, V.P. Bhallamudi, D.V. Pelekhov, F.Y. Yang, and P.C. Hammel, *Microscopic studies of nonlocal spin dynamics and spin transport.* Journal of Applied Physics, **117**(17): p. 172604 (2015)
5. S.A. Manuilov, C.H. Du, R. Adur, H.L. Wang, V.P. Bhallamudi, F.Y. Yang, and P.C. Hammel, *Spin pumping from spinwaves in thin film YIG.* Applied Physics Letters, **107**(4): p. 042405 (2015)
6. A.J. Berger, M.R. Page, J. Jacob, J.R. Young, J. Lewis, L. Wenzel, V.P. Bhallamudi, E. Johnston-Halperin, D.V. Pelekhov, and P.C. Hammel, *A versatile LabVIEW and field-programmable gate array-based scanning probe microscope for in operando electronic device characterization.* Review of Scientific Instruments, **85**(12): p. 123702 (2014)
7. A.J. Berger, M.R. Page, H. Wen, K.M. McCreary, V.P. Bhallamudi, R.K. Kawakami, and P.C. Hammel, *Correlating spin transport and electrode magnetization in a graphene spin valve: Simultaneous magnetic microscopy and non-local measurements.* Applied Physics Letters, **107**(14): p. 142406 (2015)

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10. H.J. Mamin, R. Budakian, B.W. Chui, and D. Rugar, *Detection and manipulation of statistical*

polarization in small spin ensembles. Physical Review Letters, **91**(20): p. 207604 (2003)
11. R. Schirhagl, K. Chang, M. Loretz, and C.L. Degen, *Nitrogen-Vacancy Centers in Diamond: Nanoscale Sensors for Physics and Biology*, in *Annual Review of Physical Chemistry, Vol 65*, M.A. Johnson and T.J. Martinez, Editors. 2014. p. 83-105.
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