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200 University Avenue West

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RPPR Final Report
as of 16-Sep-2021

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INVESTIGATOR(S):

Name: Raffi Budakian
Email: rbudakian@uwaterloo.ca
Phone Number: 5198884567ext
Principal: Y

Organization: **University of Waterloo**
Address: 200 University Avenue West, Waterloo, Ontario, N2L 3G1
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Begin Performance Period: 15-May-2016
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Report Term: 0-Other
Submitted By: Raffi Budakian
Email: rbudakian@uwaterloo.ca
Phone: (519) 888-4567ext
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STEM Degrees: 4
STEM Participants: 3

Major Goals: The goal of the proposed research program is to achieve nanometer scale imaging of electron and nuclear spins using silicon nanowire baed force-detected magnetic resonance spectroscopy. In particular, we seek to extend the spectroscopic capabilities of magnetic resonance spectroscopy to the nanometer scale to study the structure and function of complex biomolecules, including proteins and virus particles with high spatial resolution, in a chemically selective manner. To achieve these goals, we need to develop new techniques capable of implementing high fidelity spin control in nanometer scale spin ensembles. In addition, we need to increase the spin detection sensitivity to be able to image spin distributions with characteristic volumes of (50-nm)^3 in three dimensions with sub nanometer resolution. In this proposal, we are following two parallel routes. First, we are are designing pulse sequences using optimal control theory to implement high fidelity spin control. These pulses will then be incorporated into dynamical decoupling sequences that increase spin coherence times for high resolution imaging and spectroscopy of solid-sate nuclear spins. Second, we are increasing the detection sensitivity by (1) Establishing efficient dynamic nuclear polarization protocols for producing large nuclear spin polarization for nanometer scale imaging and spectroscopy (2) Fabricating high quality silicon nanowire arrays to optimize detection sensitivity and increase measurement efficiency. (3) Fabrication of current focusing field gradient sources using epitaxial metal films that maximize the magnetic field gradient for imaging.

Accomplishments: Please refer to the uploaded document.

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Training Opportunities: Through the support of the ARO grant, the following individuals were awarded degrees

1. Xudong Liu, MS Physics, U. Waterloo, 2018
2. Andrew Jordan, MS Physics, U. Waterloo, 2019
3. Seyed Sahand Tabatabaei, MS Physics, U. Waterloo, 2019
4. William Rose, PhD Physics, U. Illinois at Urbana Champaign, 2021

William Rose (PhD student) conducted the recently published work on high resolution nanoscaleNMR spectroscopy. In the course of this work, he gained extensive theoretical background on average Hamiltonian theory, quantum control, and implementing these concepts using custom electronics, which he designed and constructed. He was the first author on the high-profile paper, which he co-authored along with a fellow student Holger Haas, that presented these results. William is now heading the effort to conduct the NMR diffraction measurements.

5. Andrew Jordan (MSc student) is being trained to use our CVD system to grow silicon nanowireresonators. Andrew is also being trained in the cleanroom to use the deposition and lithography tools. Andrew's project will be study mechanical dissipation in single-crystal silicon resonators as a function of size, surface passivation and temperature.

6. Sahand Tabatabaei (former MSc student, current PhD student) leads the theoretical effort in developing Hamiltonian engineered NMR pulse sequences for high resolution spectroscopy and imaging.

1. Ben Yager (senior laboratory assistant) has designed a low temperature MRFM system to operate at 0.3 K, andexcite electron spins at 33 GHz. Ben has also been essential in running a CVD system for growing silicon nanowire mechanical resonators. Ben along with William Rose are heading the implementation of the NMR diffraction measurements.

The following postdoctoral fellows were supported on the

1. Michele Piscitelli - Led the effort in the nanofrabrication current focusing field gradient source devices used to generate magnetic fields used for spin detection and spin control. Her most notable contribution has been the fabrication of field gradient devices which will be used for the upcoming 3D NMR diffraction measurements of protein crystals.

2. Pardis Sahafi: Led the effort for the CVD growth of SiNWs for all of the experimental work.

3. Holger Haas - developed coherent control protocols for NMR imaging and NMR diffraction. He was the chief scientist who oversaw these critical projects.

The following undergraduate students were involved with this research

1. Namanish Singh: During his co-op term, Namanish wrote an implementation of a optimal controller for controlling the quality factor of a nanomechanical oscillator, as well as an implementation of a phase-locked loop, using an FPGA to control a 100-MHz transceiver module.

2. Kaleb Domenico Ruscitti: Kaleb Ruscitti was an undergraduate student who worked in our group for one semester as aco-op student. Kaleb helped develop a calibration procedure, based on force detection, capable of measuring the amplitude and phase of the electric currents flowing through the current-focusing field gradient source devices at the location of the spins at microwave frequencies. The technique relies upon the nonlinear coupling between the electric fields produced by currents flowing through the device and a micron-scale silicon cantilever that is placed within the detection volume. This calibration procedure established a means to characterize the frequency dependent response of the control electronics at radio and microwave frequencies. Kaleb's role: (1) Design and construct a microwave circuit to validate the calibration procedure. (2) Integrate the test device into a customized room temperature atomic force microscope. (3) Analytically derive mathematical expressions that relate the frequency dependent response of the microwave electronics to the measured signal. (4) Use COMSOL finite element simulation software to theoretically model the coupling between the electric currents and the cantilever.

3. Michael Wolf: Michael Wolf was a summer student who worked on developing the electronics for the 33-GHz IQ modulator used for controlling electron spins. His main task was to assemble and validate the performance of the chain of modulators, filters and amplifiers used to generate the ESR waveforms used for controlling electron spins.

Results Dissemination: The results that were generated through the support from this ARO grant were presented in several international conferences.

1. Spin Mechanics 5 and Nano MRI 6 workshop hosted by Ecole de Physique Des Houches, 2018.

2. NanoMRI 7 workshop held virtually at the Weizmann Institute for Science, Israel, April 2020.

3. The talk was also given at various seminars, colloquia and workshops held in US and Canada.

Honors and Awards: In 2019, I was granted the Waterloo Institute for Technology / Institute for Quantum Computing Endowed Chair in Nanotechnology for my work in nanometer scale magnetic resonance imaging.

Protocol Activity Status:

Technology Transfer: During the grant cycle, we applied for and received a patent for nuclear magnetic resonance diffraction. The IP explains a method for atomic scale imaging of materials with atomic resolution.

R. Budakian ,H. Haas, and Quantum Valley Investment, “Nuclear Magnetic Resonance Diffraction,” US Patent No.: 10,585,154, March 10, 2020.

PARTICIPANTS:

Participant Type: Postdoctoral (scholar, fellow or other postdoctoral position)
Participant: Michele Piscitelli
Person Months Worked: 6.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Graduate Student (research assistant)
Participant: Seyed Sahand Tabatabaei
Person Months Worked: 6.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Graduate Student (research assistant)
Participant: Xiudong Liu
Person Months Worked: 6.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Graduate Student (research assistant)
Participant: Andrew Jordan
Person Months Worked: 6.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Undergraduate Student
Participant: Michael Wolf
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Undergraduate Student
Participant: Namanish Singh
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Graduate Student (research assistant)
Participant: William Rose
Person Months Worked: 12.00 **Funding Support:**
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Article Title: High-Resolution Nanoscale Solid-State Nuclear Magnetic Resonance Spectroscopy
Authors: William Rose, Holger Haas, Angela Chen, Nari Jeon, Lincoln Lauhon, David Cory, Raffi Budakian
Keywords: Nanoscale magnetic resonance imaging, nuclear magnetic resonance, force detection, quantum control, nanowires
Abstract: We present a new method for high-resolution nanoscale magnetic resonance imaging (nano-MRI) that combines the high spin sensitivity of nanowire-based magnetic resonance detection with high-spectral-resolution nuclear magnetic resonance (NMR) spectroscopy. Using a new method that incorporates average Hamiltonian theory into optimal control pulse engineering, we demonstrate NMR pulses that achieve high-fidelity quantum control of nuclear spins in nanometer-scale ensembles. We apply this capability to perform dynamical decoupling experiments that achieve a factor of 500 reduction of the proton-spin resonance linewidth in a (50?nm)^3 volume of polystyrene. We make use of the enhanced spin coherence times to perform Fourier-transform imaging of proton spins with a one-dimensional slice thickness below 2 nm.
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Article Title: Ultralow Dissipation Patterned Silicon Nanowire Arrays for Scanning Probe Microscopy
Authors: Pardis Sahafi, William Rose, Andrew Jordan, Ben Yager, Michèle Piscitelli, Raffi Budakian
Keywords: Mechanical resonator, SiNW, silicon, nanowire, SPM, force detection
Abstract: In recent years, self-assembled semiconductor nanowires have been successfully used as ultrasensitive cantilevers in a number of unique scanning probe microscopy (SPM) settings. We describe the fabrication of ultralow dissipation patterned silicon nanowire (SiNW) arrays optimized for scanning probe applications. Our fabrication process produces ultrahigh aspect ratio vertical SiNWs that exhibit exceptional force sensitivity. The highest sensitivity SiNWs have thermomechanical noise limited force sensitivity of 9.7 ± 0.4 aN/ Hz at room temperature and 500 ± 20 zN/ Hz at 4 K. To facilitate their use in SPM, the SiNWs are patterned within 7 ?m from the edge of the substrate, allowing convenient optical access for displacement detection.
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Article Title: Numerical Engineering of Robust Adiabatic Operations
Authors: Sahand Tabatabaei, Holger Haas,William Rose, Ben Yager, Michele Piscitelli, Pardis Sahafi, Andrew Jo
Keywords: quantum control, quantum information processing, nuclear magnetic resonance
Abstract: Adiabatic operations are powerful tools for robust quantum control in numerous fields of physics, chemistry, and quantum information science. The inherent robustness due to adiabaticity can, however, be impaired in applications requiring short evolution times. We present a single versatile gradient-based optimization protocol that combines adiabatic control with effective Hamiltonian engineering in order to design adiabatic operations tailored to the specific imperfections and resources of an experimental setup. The practicality of the protocol is demonstrated by engineering a fast, 2.3 Rabi cycle-long adiabatic inversion pulse for magnetic resonance with built-in robustness to Rabi field inhomogeneities and resonance offsets. The performance and robustness of the pulse is validated in a nanoscale force-detected magnetic resonance experiment on a solid-state sample, indicating an ensemble-averaged inversion accuracy of 99.997%. We further showcase the utility of our protocol by providi
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Authors: William, Rose
Acknowledged Federal Support: **Y**

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Authors: Xudong Liu
Acknowledged Federal Support: **N**

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Authors: Andrew Jordan
Acknowledged Federal Support: **N**

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Title: Numerical Engineering of Adiabatic Quantum Operations
Authors: Seyed Sahand Tabatabaei
Acknowledged Federal Support: **N**

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Patent Country: USA
Application Date: 29-Jan-2018 Application Status: 3
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Patent Abstract:
Patent Number: 10,585,154
Patent Country: USA
Application Date: 28-Jan-2018 Application Status: 3
Date Issued: 10-Mar-2020

Partners

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RPPR Final Report
as of 16-Sep-2021

I certify that the information in the report is complete and accurate:
Signature: Raffi Ohannes Budakian
Signature Date: 5/26/21   4:15PM

Through the support of ARO grant W911NF1610199, during the period May 15, 2016 to Aug. 14, 2020 there were 4 major accomplishments that have significantly advanced the capabilities for force-detected imaging of nuclear spins on the nanometer scale. These accomplishments are listed below.

1. **High-resolution nanometer scale solid-state nuclear magnetic resonance spectroscopy:** We present a new method for high-resolution nanoscale magnetic resonance imaging (nano-MRI) that combines the high spin sensitivity of nanowire-based magnetic resonance detection with high-spectral-resolution nuclear magnetic resonance (NMR) spectroscopy. Using a new method that incorporates average Hamiltonian theory into optimal control pulse engineering, we demonstrate NMR pulses that achieve high-fidelity quantum control of nuclear spins in nanometer-scale ensembles. We apply this capability to perform dynamical decoupling experiments that achieve a factor of 500 reduction of the proton-spin resonance linewidth in a 50-nm^3 volume of polystyrene. We make use of the enhanced spin coherence times to perform Fourier-transform imaging of proton spins with a one-dimensional slice thickness below 2 nm.

2. **Ultralow dissipation patterned silicon nanowire arrays for scanning probe microscopy:** Access to high quality nanowire mechanical resonators is essential to ultra-sensitive force-detected magnetic resonance imaging. During the period from August 1, 2018 to July 31, 2019, we focused on the fabrication of ultra-sensitive silicon nanowire (SiNW) arrays for high spin sensitivity magnetic resonance force microscopy (MRFM) measurements. We developed a robust process for fabricating SiNW arrays that exhibit ultra-low mechanical dissipation, with controllable geometry, that are tailored for use in MRFM, and other scanning probe microscopy (SPM) applications that demand the highest force sensitivity. Fig. 1 shows a schematic, along with optical and SEM images, of the fabricated SiNW devices. Two different diameter SiNW arrays were investigated: Array 1 (132-nm diameter, 23-μm long) and Array 2 (77-nm diameter, 23-μm long). We characterized the mechanical properties of a large number of SiNWs for each array as a function of temperature from 295 K to 4 K, and found that the frequency, spring constant, and quality factor of individual SiNWs were highly consistent within a given array.

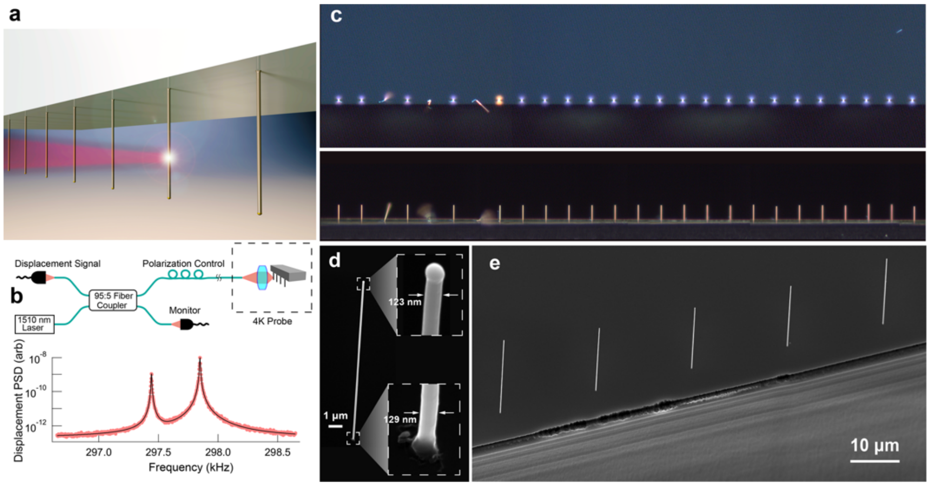


Figure 1. (a) Rendering of a SiNW array with displacement detection laser. (b) Top: schematic diagram of the interferometer. Bottom: characteristic power spectral density of thermal oscillations of the two fundamental NW flexural modes. (c) Optical image of SiNWs, from array 1, grown at the substrate edge viewed from above (top photo) and from the side edge on (bottom photo). Each dot in the top photo represents a vertical SiNW. (d) SEM of a single SiNW with insets showing the diameter at the tip and base. (e) SEM of vertical SiNWs at the sample edge.

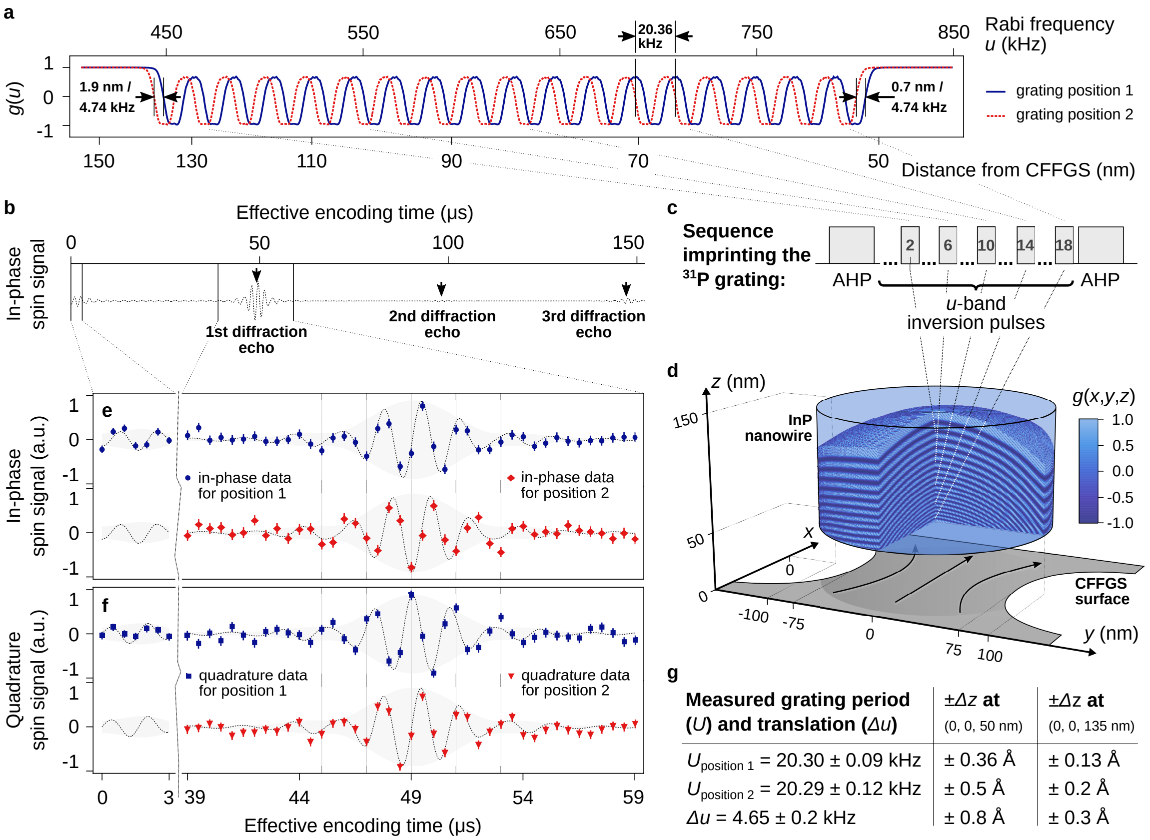
Our fabrication process produces vertical SiNWs with highly consistent mechanical properties, extremely low mechanical dissipation, and ultra-high force sensitivity. The highest sensitivity SiNW's have thermomechanical noise-limited force sensitivity of 9.7 ± 0.4 aN/Hz$^{1/2}$ at room temperature and 500±20 aN/Hz$^{1/2}$ at 4 K. For comparison, a single electron spin placed in a magnetic field gradient of 10^6 T/m will produce a peak force of 9.3 aN, which is approximately equal to the 9.7 aN-RMS force noise in the 1-Hz bandwidth of the 77-nm diameter SiNWs at room temperature. The same diameter SiNWs would be capable of detecting 0.5 aN-RMS in a 1-Hz bandwidth at 4 K, equal to just 51 proton spins in a peak field gradient of 10^6 T/m. For reference, the current-focusing field-gradient source (CFFGS) devices developed through the support of ARO, are capable of generating time dependent field gradients equal to 10^6 T/m. In this grant cycle, we have developed a platform that combines ultra-high force sensitivity mechanical sensors with intense time dependent magnetic field gradients, and Hamiltonian engineered NMR pulses for coherent spin control. These advances pave the way for atomic scale magnetic resonance imaging of molecular assemblies. In the coming months, the SiNW arrays will be used for 3D NMR imaging and atomic scale NMR diffraction measurements.

3. **Numerical engineering of robust adiabatic operations:**

Adiabatic operations are powerful tools for robust quantum control in numerous fields of physics, chemistry and quantum information science. The inherent robustness due to adiabaticity can, however, be impaired in applications requiring short evolution times. We developed a single versatile gradient-based optimization protocol that combines adiabatic control with effective Hamiltonian engineering in order to design adiabatic operations tailored to the specific imperfections and resources of an experimental setup. The practicality of the protocol is demonstrated by engineering a fast, 2.3 Rabi cycle-long adiabatic inversion pulse for magnetic resonance with built-in robustness to Rabi field inhomogeneities and resonance offsets. The performance and robustness of the pulse is validated in a nanoscale force-detected magnetic resonance experiment on a solid-state sample, indicating an ensemble-averaged inversion accuracy of 99.997%. The development of fast adiabatic operations is an essential element to the spin detection protocol. The ability to perform high fidelity fast adiabatic inversions of spins allows us to perform spin detection that better evade frequency noise that the SiNW force sensor experiences near the surface, and thus permits more sensitive spin detection.

4. **Nuclear magnetic resonance diffraction:**

In 1973 Mansfield and Grannell published a paper titled "NMR 'diffraction' in solids?", where they analyzed and experimentally demonstrated the equivalence between x-ray diffraction and the underlying principles of Fourier imaging in nuclear magnetic resonance. Nevertheless, there has been no demonstration of nuclear magnetic resonance diffraction (NMRD) that was initially considered by Mansfield and Grannell, as it requires extremely intense uniform magnetic field gradients across a single crystal lattice of nuclear spins. While the magnetic field gradients necessary for performing NMRD are inaccessible for sample dimensions typical of nuclear magnetic resonance, they can be generated for sample volumes around 100-nm^3. During this grant cycle, we performed one-dimensional NMRD experiment performed on a nanometer-scale modulation of nuclear spin state in a single-crystal nanowire sample. We generated a “diffraction grating” in an ensemble of ^{31}P spins in an InP nanowire by modulating the spin state through repeated selective spin inversions for a sequence of sample regions the linear dimension of which ranges between 1.6 nm and 3.7 nm. Contrary to most diffraction techniques, spin signal in magnetic resonance can be collected with full phase information, hence, the spin sample geometry and the absolute position of the diffracting spins can be inferred from NMRD signal directly through the Fourier transform. We demonstrate both of these features of NMRD in our experiments, and measure the periodicity as well as a translation in spin state modulation with sub-Angstrom precision. Fig.2 shows the experimental protocol, and the NMRD diffraction data. Two gratings were encoded into the spin system; the absolute position of the gratings was determined with an accuracy of ±0.4 Å, and the relative position between the two gratings was determined with an accuracy of ±0.2 Å. This is the highest resolution Fourier transform magnetic resonance imaging performed. We have received the US patent 10,585,154 titled “Nuclear Magnetic Resonance Diffraction,” which presents a platform for applying the NMRD to image nuclear spins with atomic resolution in nanometer scale structures that possess a periodic structure. Ongoing work in the group is focused on applying NMRD to image proton spins in protein nanocrystals. At the moment, there are no well-established methods for imaging hydrogen atoms in proteins, as protons are invisible to X-rays. We believe NMRD is well-suited to address this problem, as proton spins yield the largest NMR signal. The ability to image proton coordination in proteins has been identified as an important capability, that is currently lacking. One of the current projects in the group is focused on the synthesis of protein nanocrystals and to apply NMRD to image proton cooridination.



Measured grating period (U) and translation (Δu)	$\pm\Delta z$ at (0, 0, 50 nm)	$\pm\Delta z$ at (0, 0, 135 nm)
$U_{\text{position 1}} = 20.30 \pm 0.09$ kHz	± 0.36 Å	± 0.13 Å
$U_{\text{position 2}} = 20.29 \pm 0.12$ kHz	± 0.5 Å	± 0.2 Å
$\Delta u = 4.65 \pm 0.2$ kHz	± 0.8 Å	± 0.3 Å

Figure 2: (a) Schematic of the NMR diffraction grating. The diffraction is encoded in the Rabi frequency domain, and has a period of 20 kHz. The top axis shows the range of Rabi frequencies that was used to encode the 18-period grating. The bottom axis shows the range of positions along the z-axis spanned by the grating. (b) The calculated NMRD signal based on the sample geometry. (c) Schematic of the NMR pulse sequence used for encoding the grating. (d) The figure shows the measured signal and comparison to the expected NMRD signal for the in-phase and quadrature components of the spin signals. (g) The period and position of the encoded grating determined using the NMRD technique.