Scanned Probe Magnetic Resonance Force Microscopy

CNF Project Number: 863-00
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Abstract:

Magnetic resonance force microscopy (MRFM) is a scanned-probe technique conducted at cryogenic temperatures to detect the spin density of a sample as a force exerted on a high-compliance cantilever. This 3D-nanoscale imaging technique offers a promising scope of applications, ranging from imaging of biological molecules to characterizing buried interfaces in organic and hybrid semiconductor devices. MRFM has already demonstrated the 20-30 nm depth of view required for imaging macromolecules and macromolecular complexes. However, to capitalize on the depth of view demonstrated by MRFM, we must operate at small tip-sample separations. This comes at a cost of larger surface-induced dissipation, resulting in decreased force sensitivity. In order to improve our spin detection sensitivity, we need a better understanding of the fundamental limits underlying surface-induced dissipation. Accordingly, our work at the Cornell NanoScale Science & Technology Facility (CNF) this year built upon prior work and extended to initiating new fabrication protocols that will enable a deeper understanding of cantilever dissipation and frequency noise.

Summary of Research:

High sensitivity in MRFM is reached by studying a small number of spins-down to ultimately single spin-at a time. Sensitivity is governed by two limiting factors. In the first limit, sufficiently large tip-field gradients are required for optimal MRFM detection as signal is detected through spin interactions with the field gradient of a magnetic particle. Prior work in the Marohn group demonstrated the successful fabrication of 200 nm wide high-gradient nanomagnets on cantilevers for scanned probe detection of magnetic resonance via a batch and serial-fabrication approach [1]. We have successfully extended this electron-beam lithography-based protocol to fabricate 50 nm wide cobalt nanomagnets suitable for use in a magnetic resonance force microscope [2]. Achieving this size reduction required careful revision of the resist preparation and identification of the correct electron-beam dose. The anticipated factor of four increase in tip-field gradient is expected to translate into a 16-fold reduction in acquisition time in the polarized-spin limit and a 256-fold reduction in acquisition time in the unpolarized-spin limit. These magnets will be used in experiments designed to determine the magnet dimensions and compositions yielding the largest possible tip-field gradient and — accordingly — maximizing the spin force.

In the second limit, noncontact dissipation between the tip and the sample surface must be minimized; the observed dissipation remains the biggest factor limiting our detection sensitivity. There are two general dissipation mechanisms to consider: (1) tip charge coupling to fluctuating electric field gradients, and (2) tip magnetization coupling to fluctuating magnetic field gradients in the substrate, equivalent via the fluctuation-dissipation theorem to eddy current damping. To test the second mechanism, fluctuations experienced by a nanomagnet-tipped
cantilever over a patterned sample surface will be studied. To pattern the sample, a shadow mask protocol was implemented. The shadow mask was fabricated starting with a double-sided polished Si wafer. Two microns of GSI PECVD oxide were deposited front and back. The front side was patterned by contact photolithography, followed by an oxide etch then a through-wafer BOSCH Si etch. The leftover oxide was finally released in a buffered oxide etch (BOE). The sample was then patterned via four serial electron beam evaporations as shown in Figure 2.

The experimental outcome of our fluctuation studies, combined with a unified theoretical picture of viable dissipation mechanisms, will help direct modifications in tip fabrication as well as sample preparation to improve sensitivity in MRFM particularly and scanning probe microscopy (SPM) generally.

References:
Micro-Tweezers for the Free Manipulation of Carbon Nanotubes and Graphene

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Abstract:
Electrically-contacted micro-tweezers provide a unique platform for precision positioning and mechanical and electrical measurements of carbon nanotubes (CNTs) and graphene. Using these micro-tweezers, we demonstrate the coupling of small-diameter CNTs to a high-finesse optical cavity, which permits low-noise measurements of the thermal resonance modes of the CNTs. We also demonstrate the free manipulation of a graphene device while maintaining electrical contact to it.

Summary of Research:
The micro-tweezers are made from two components: gold for the tweezer tips, and SU-8 as a supporting, semi-flexible frame. Electroplated gold (Au) ~ 1 µm thick is used to form the tweezer tips, which both mechanically support and electrically contact the CNTs and graphene devices. A ~ 160 µm thick SU-8 frame is patterned over the gold to support the micro-tweezers themselves, as is shown in Figure 1a. While the flexibility and ease of thick-film patterning of SU-8 make it a particularly effective structural support, SU-8 adheres poorly to Au [1]. To remediate this issue, Au “dendrites” tens of microns in length were grown via electroplating at ~ 100 mA in various regions along the tweezer electrodes. SU-8 was then spun and baked on over the dendrites, allowing them to stick into the SU-8 frame and yielding significantly improved adhesion. Although measurement of the binding strength has not been attempted, tweezers without dendrites failed to adhere to the SU-8, whereas tweezers with dendrites on the same wafer remained attached. An example of dendrite growth is shown in Figure 1b.

As a demonstration of the functionality of these micro-tweezers, we used them to couple CNTs to the evanescent fields of Si3N4 microdisk optical resonators, which were fabricated by our collaborators in Michal Lipson’s research group [2]. Laser light is coupled from a tapered optical fiber into the microdisk, and the circular geometry of the microdisk results in steady-state optical resonance modes known as whispering gallery modes. The outgoing light from the cavity is collected and measured by a photodiode. A schematic of the experimental setup is shown in Figure 2.
Initial coupling between the CNT and the optical cavity is detected by tuning through cavity resonance with and without a nearby CNT. The transmitted power shows resonance broadening and decreased extinction on resonance in the presence of a CNT, as shown in Figure 3a. The micro-tweezers precisely move the CNT toward and away from the microdisk, allowing us to find the region of highest transmission to height sensitivity. The power spectrum of the photodiode is measured and used to calculate a displacement power spectrum. Five modes are detected with a noise floor of 30 pm/√Hz as shown in Figure 3b. The ability to optically detect thermal resonances with low noise is not only an improvement on previously reported electronic measurements, but also provides an accessible measurement technique for optical cooling and parametric amplification.

We have also begun using the micro-tweezers to manipulate graphene devices, as shown in the inset of Figure 4. We pattern graphene with gold contacts onto an aluminum release layer so that the devices can be freely manipulated by the tweezers in water. Figure 4 shows that the micro-tweezers maintain electrical contact with a graphene device over a range of successive movements, from (1) initial contact on the substrate, to (2) lifting the device up into the water, and (3) stretching and (4) compressing it. Throughout this motion, during which the liquid-gate voltage (VLG) of the water is held at 0 V, the source-drain current (ISD)-voltage (VSD) response of the device remains virtually unchanged.

The ability to controllably manipulate and electrically contact CNTs and graphene using our homebuilt micro-tweezer apparatus permits exploration of a variety of phenomena that were previously difficult to access. The CNT microdisk resonator experiments presented here show promise for continued work in opto-mechanical systems with nanotubes, and the ability to maneuver graphene devices off the substrate without losing electrical contact provides a new route to electrical sensing experiments using graphene.

**References:**


Silicon Optomechanical Transducer

CNF Project Number: 1380-05
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Abstract:
We report the fabrication of silicon optomechanical resonators on the silicon-on-insulator (SOI) platform.

Summary of Research:
The silicon resonator discussed here was designed for optical sensing of mechanical motion of specific modes of the resonator. The fabrication process flow is shown in Figure 1. We start with a SOI wafer and grow thermal oxide that will act as a hard mask. Next, we do e-beam lithography in the JEOL 9500 to define the structure. The 9500 is necessary to achieve features < 100 nm over a write field of 1 mm. We write an 8X6 array of write fields on a 2X2 cm chip allowing us to fabricate many devices in one fabrication run. This is followed by an etch to define the hard mask in the Oxford 81 followed by device layer silicon etch in the PT770 ICP anisotropic silicon etcher. Finally, we pattern a release window in photoresist using the ABM contact aligner and etch the underlying oxide with BOE.

The final step is critical point drying in the Leica CPD to release the device. Figure 2 is an SEM of the final suspended device. The resonator is a double ring structure so as to isolate the electrostatics from the optics.

References:

The Nanoaquarium –
A Device for in situ Electron Microscopy of Processes in Liquids

CNF Project Number: 1542-07
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Abstract:
The nanoaquarium is a nanofluidic platform for in situ electron microscopy of processes in liquid media. The nanoaquarium consists of a hermetically sealed thin (~100 nm tall) liquid cell sandwiched between two electron-transparent silicon nitride membranes. The nanoaquarium is equipped with micro patterned electrodes. The device has been used to image electrochemical deposition and etching, growth and dissolution of nanoparticles, crystallization, nucleation and bubble growth, and interfacial phenomena, to assess the interactions between electron radiation and fluids, and to pattern nanostructures without a need for a mask.

Figure 1: A photograph of the nanoaquarium (top) and a schematic of the nanoaquarium’s cross-section (bottom).

Summary of Research:
The nanoaquarium is a microfabricated, nanofluidic device for in situ electron microscopy of processes taking place in liquids and of objects submerged in liquids (Figure 1). Two thin, electron-transparent silicon nitride membranes sandwich a thin liquid layer, ranging in thickness from tens of nanometers to a few microns. The cell is hermetically sealed from the vacuum environment of the electron microscope. Due to its small thickness, the liquid layer does not scatter electrons extensively and allows high resolution imaging of objects suspended in liquid. The nanoaquarium contains electrodes patterned on the silicon nitride membrane for electrochemical experiments.

The fabrication of the device was accomplished with direct bonding of silicon wafers coated with silicon nitride. One of the wafers contains a thin film of patterned silicon oxide that defines the shape and height of the imaging chamber and liquid supply conduits. The use of direct wafer bonding eliminates risk from potential contamination from glue, epoxy, and other sealing materials and assures a hermetic seal. Use of a dielectric material as the spacer allows electrodes to be directly integrated into the device. A schematic of the nanoaquarium is depicted in Figure 1. The fabrication process has been previously described [1]. Our lab, in collaboration with others, has used nanoaquariums to study nanoparticle aggregation and colloidal crystal...
growth dynamics [2-4]; interactions of nanoparticles with moving interfaces [5]; electron beam-induced radiolysis [13-14]; bubble nucleation, growth and detachment [6,10,12]; crystallization [8]; and electrochemistry [9,11]. We have also demonstrated the use of the electron beam as a “pen” to pattern nanowires without a need for a mask [12] (Figure 2).

Our most recent studies with the nanoaquarium have focused on modeling the interactions between the electron beam and water (radiolysis) [13]. The radiolysis products include, among other things, e⁻ (solvated electrons), H₂, H₂O₂, OH, and O₂. Significantly, under continuous irradiation, the concentrations of the radiolytic products do not increase unabated with time, even under the high dose rate of the electron beam, but reach rapidly equilibrium concentrations. At high irradiation dose rate, the concentrations of the gaseous species exceed supersaturation levels and bubbles nucleate, grow, and migrate [12]. The nanoaquarium provides a unique means to examine nucleation and growth of nanobubbles. A good understanding of electron beam effects is critical to correctly interpret electron microscope observations and to mitigate and exploit beam effects [13-14]. Under various dose rates, the electron beam can be used to either etch crystals or reduce and precipitate cations in solution.

Acknowledgements:

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References:

MEMS Vibration Energy Harvester

CNF Project Number: 2074-11
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Abstract:

Electrostatic power generators employ micro-fabricated variable gap capacitors to harvest mechanical vibration energy and have applications in wireless sensors networks. Prototype designs with in-plane gap-closing topology and nonlinear springs were fabricated on silicon-on-insulator (SOI) wafers with a 200 µm device layer of doped silicon (0.01 Ω cm), 3 µm oxide layer, and 350 µm handle layer. The handle layer is etched away under the electrodes and spring beams, but remains attached underneath the central area of mobile electrode to increase the shuttle mass. A DRIE etch with an aspect ratio of 20 is achieved with optimized etch parameters. A shadow mask is used for depositing parylene-C on the etched electrodes. The device is encapsulated in a clear cover to allow for visual inspection and protect from dust during experiments.

Summary of Research:

The study of microelectromechanical systems (MEMS) for vibration energy harvesting has advanced significantly in recent years. Many design features of in-plane resonant variable capacitor devices have been studied, such as the design of nonlinear springs [1], bi-stable springs [2,3], electrode geometry [4], transduction topology [5], and end stops [6]. Additionally, the behavior of such devices when excited by broadband vibrations [1], as opposed to narrowband vibrations which are far less common in ambient environments [7], is another major focus area. However, most design topologies studied are of the overlap-gap design, which has been shown to be less ideal than the gap-closing topology [8]. Fewer devices of the gap-closing topology have been fabricated and tested for vibration energy harvesting, and therefore the dynamic electrostatic behavior under power harvesting conditions should be explored.

Devices were fabricated with a five mask process that included backside processing—major process steps shown in Figure 1. One primary goal of the research was to etch the gap between electrodes at an aspect ratio 20, requiring optimization of etch parameters. Final etch profile of optimized recipe is shown in SEM images (Figure 2). The last processing step included the use of a shadow mask to deposit a conformal coating of parylene-C on the device electrodes to keep them electrically insulated from one another and to provide an impact barrier during vibration testing.

Once fabricated, the device dies are attached to a custom designed printed circuit board (PCB) with epoxy, electrically connected with wire bonding, and encapsulated with a cover made from laser cut silica and acrylic (Figure 3). The cover provides dust protection and allows for videos to be captured of the vibrating device with a high speed camera. The devices are mounted to a shaker platform oriented for in-plane vibration testing (Figure 4). Results on the latest devices are still pending.

Figure 1: The five mask fabrication process flow.
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References:


Figure 2: High aspect ratio etch profile.

Figure 3: Device on PCB with encapsulation.

Figure 4: Device on shaker setup with data acquisition and high speed camera.
Design and Fabrication of Universal TGV
Reliability Test Vehicle Using a Glass Interposer

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Abstract:
In this project, contrary to the conventional silicon, a glass wafer is used throughout our fabrication processes. Glass fabrication processes are not characterized well compared with the conventional silicon, therefore, we understand glass wafer fabrication has many risks, and unexpected results, such as reflection through the transparent glass, adhesion problem between a glass wafer and various metals, and so on. Thus, we try to approach the fabrication process as an exploring and initiating step for characterization of the glass wafer.

Summary of Research:
Introduction and Background. Glass offers not only good dimensional and thermal stability, but also potential advantages in these respects, including the ultimate scale-up to roll-to-roll processing. In addition, coefficients of thermal expansion (CTE) can be tailored to specific needs. Glass interposer may come in useful in applications where the CTE is mismatched between the die and an organic carrier or printed circuit board.

In this work, various reliability tests using designed universal through glass via (TGV) reliability test vehicles are proposed. The test vehicle contains many features addressing the risks and capabilities of in situ monitoring and heating to mimic the operating condition. Through the reliability tests, we can study not only basic physical fatigue failures but also electromigration behavior of the microbumps between chip and glass interposer. Furthermore, the lifetime of the devices can be estimated by the result of the reliability tests.

Results:
Most processes such as lithography, electroplating, and CMP can be applied to the glass wafer. However, they cannot be accepted with the directly same recipe when the glass wafer is used instead of the silicon wafer. Especially,
the transparency of a glass wafer causes a reflection problem when UV light goes through the wafer and reflects to the deposited photoresist with a longer path (summation of photoresist and glass wafer thickness) than the silicon wafer while under the same UV exposure. This makes it hard to get fine pitch patterns under 10 µm without optimization of the recipe for photolithography process. Thus, various recipes are used to make it optimize and fine pitch patterns are obtained as shown in Figure 1.

Sputtering processes have to be modified using ionized plasma to have a reliable seed layer for the electroplating process of Cu because adhesion between a glass and the conventional seed layers for Si can be different. In addition, while deep reactive ion etching (DRIE) process is commonly used to form through silicon vias or blind vias of the silicon wafer, laser drilling is conducted to make through holes of the glass wafer; blind vias cannot be fabricated on the glass wafer.

It means that we cannot adopt the matured recipe of Si wafer which is represented as blind via etching – via filling – back grinding process. Thus, its optimization is under process as shown in Figure 2.

Besides, only both sides electroplating can be applied to the glass wafer since through holes are constructed on the glass, flowing current is very important to obtain void-free via filling plating process. Since two edges from the top and bottom sides where flowing current can be concentrated should be considered to avoid making a void at the center, forward and backward current are mixed appropriately with many trials and errors.

At last, CMP process is not easy to perform since both sides are plated during the electroplating; there is no flat surface which can be a basis for the CMP. This process is planned to conduct in this summer.