MEMS-based Force-Detected Nuclear Magnetic Resonance (FDNMR) Spectrometer

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ABSTRACT

Nuclear Magnetic Resonance (NMR) spectroscopy allows assignment of molecular structure by acquiring the energy spectrum of nuclear spins in a molecule, and by interpreting the symmetry and positions of resonance lines in the spectrum. As such, NMR has become one of the most versatile and ubiquitous spectroscopic methods. Despite these tremendous successes, NMR experiments suffer from inherent low sensitivity due to the relatively low energy of photons in the radio frequency (rf) region of the electromagnetic spectrum. Here, we describe a high-resolution spectroscopy in samples with diameters in the micron range and below. We have reported design and fabrication of force-detected nuclear magnetic resonance (FDNMR).

Keywords: NMR, FDNMR, MEMS, spectrometer

1. INTRODUCTION

Nuclear magnetic resonance is a physical phenomenon that occurs when the nuclei of certain atoms are immersed in a static magnetic field and exposed to a second excitation magnetic field. Some nuclei experience this phenomenon and other do not, dependent on whether they possess a property called spin [1]. The macroscopic effect of spins of nuclei can be considered as a magnetization induced by the sample, which can be manipulated by the excitation magnetic field under certain conditions. In induction-detected nuclear magnetic resonance, the excitation magnetic filed oscillates with a specific frequency, the Larmor frequency, and a coil is used t sense the motion of the magnetization.

FDNMR is a method of mechanically detecting magnetic resonance [2-4]. The detector consists of a ferromagnet harmonically bound to a mechanically bound to a mechanical resonator and measures a magnetic force of interaction with a nearby sample via dipole-dipole coupling. Flexural modes of vibration of the resonator are induced by inversion of the sample magnetization at the mechanical resonance frequency of the device. In this method, a nominally homogeneous field at the sample allows coherent spectroscopy over the entire sample volume.

Sensitivity analyses suggest that encoding an NMR signal into mechanical oscillations favors inductive detection at the micro scale and below with Brownian motion of the detection being the predominant source of noise and eddy currents being the predominant source of damping [5]. As such, the design issues of a MEMS-based spectrometer optimized for 50 micron samples have been investigated. The microfabrication of the device will be also discussed.

2. DESIGN

It concerns the design issues of a MicroElectroMechanical System (MEMS) based force-detected NMR spectrometer. As a means of directing the steps towards microfabrication, we wish to consider structures with: 1) narrow, unobstructed detector/annulus magnet gaps to maintain high field homogeneity, 2) low stress electroplated magnetic films, 3) mechanical oscillator structures with low dissipation and inertial mass, 4) fine control over observed resonance frequencies, 5) a means of mitigating damping due to eddy currents, and 6) low power and portability to allow both terrestrial and laboratory spectroscopic studies.

General Design Issues

The mechanical oscillator should be designed such that the inertial mass is small compared to that of the detector magnet, with low mechanical dissipation, high torsional rigidity to counteract the torsional stress place on the detector magnet in the annulus. Of particular importance is the balancing of the resonator's elastic k_{elas} and magnetic k_{mag} spring constants. k_{elas} is due to the restoring force of the beam, and k_{elas} is due to magnetic forces between the detector and annulus. The magnetic spring constant is negative and will soften the effective spring constant $k_{eff} = k_{elas} + k_{mag}$. Therefore, k_{eff} will depend upon the magnetic field strength, the relative sizes of the magnets, and the gap between the detector and annulus.

We also seek a device with a resonance frequency in a ~ 2T magnetic field of 1-10 kHz in order to efficiently invert the spin magnetization with adiabatic rapid passage. The ~ 2T field is chosen based upon the use of soft and low stress magnetic materials that can be readily fabricated into a deice with saturation magnetization between 1.6 – 2.0 T/μ_o and the restriction of the use of hard permanent magnets to provide the static field. The restriction is based upon the goal of a portable, low-power spectrometer, where we have restricted the total power to be less than 100 μW and the majority of this power will be utilized by the radiofrequency excitation circuit.

3. MICROFABRICATION

a. *(3:1) aspect ratio "sunshine" thick photoresist mold*: Preliminary photoresist mold used to create the annulus-detector magnet assembly. The "sunshine" pattern was used to create the annular radial slits to reduce eddy current damping [6]. The gap size is 3 μm and the slits are 3 μm x 3 μm.

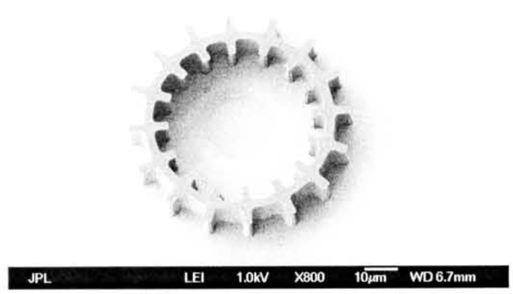

Figure 1. SEM image of microfabricated "sunshine" photoresist mold.

b. *Electroplated soft magnet*: A goal of ours is to have a low-stress magnetic material for use in the detector and annular magnets. The magnetic material should have a *Ms* of 1.6 - 2.0 T/ μ_o and have good resistance to corrosion by the buffered oxide etch (BOE) used during microfabrication. Electrodeposition allows specifically patterned magnets to be incorporated onto the mechanical oscillator structures and may allow feature sizes below 1 μm to be incorporated for creation of the detector annular gap and radial slits to mitigate eddy current damping by the use of appropriate photoresist molds. The alloy composition is controlled through adjusting bath salt and additive concentration. Typically, materials with high Ms have very high stress, greater than 250 MPa, which will likely result in delamination of the film. Therefore, we seek materials with internal stresses of less than 80 MPa. Experimentally, we have observed that 85/15 Co:Ni films have saturation magnetizations of 1.6 T/ μ_o with internal stresses of 65MPa.

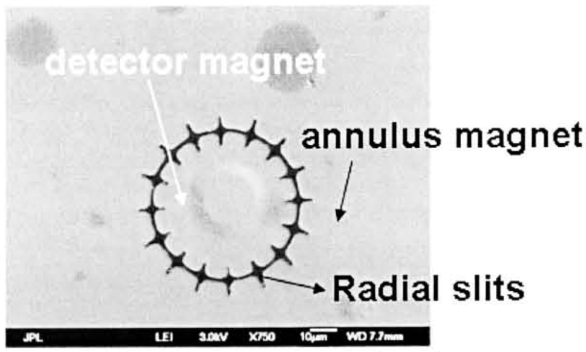

Figure 2. SEM image of electroplated detector and annulus magnets.

c. *Mechanical Si resonator*: The mechanical oscillator should be designed in a way to constrain the detector magnet motion to the z-axis, with torsional rigidity to prevent the detector magnet to torque in its bore, be constructed of a low-dissipation material that relatively easy to use in standard microfabrication processes, and counteract the negative magnetic spring constant. A natural choice of a material is silicon, given the explosive volume of known microfabrication methods and its inherent low dissipation leading to high sensitivity to resonant driving as well as its relatively large Young's modulus.

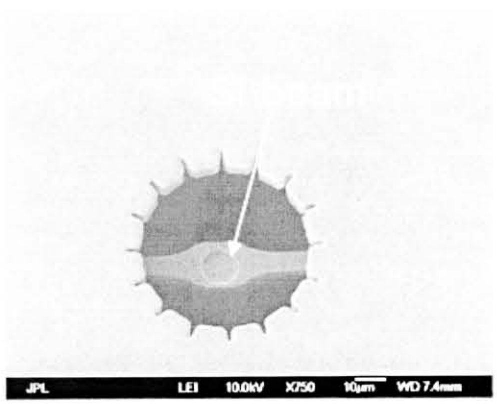

Figure 3. SEM image of silicon resonator underneath the detector magnet.

d. *Lateral interferometer*: Fiber-optic interferometry (FOI) is a versatile method for displacement sensing of both micro- and macro-scale objects. One of the strongest motivations for using FOI for displacement sensing is its excellent sensitivity with larger dynamic range. If the fiber-optic interferometer were to operate in its normal longitudinal arrangement, where the fiber core sits directly above the measured oscillator, the hole required through the static field magnets would impart a severe reduction in magnetic field strength and would introduce large field inhomogenity across the sample. An alternative is to cleave the end of the fiber at 45^o such that the fiber axis is parallel to the annular and detector magnets and the long axis of the oscillator. In this arrangement, we have a transverse, fiber-optic interferometer. We have made a trench for the optical fiber to be located above the detector magnet.

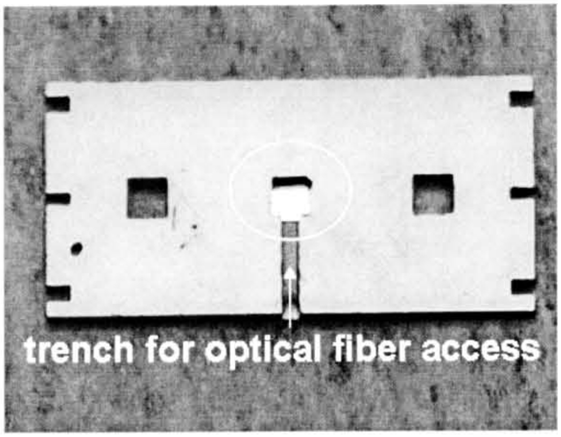

Figure 4. Microphotograph of the bottom of the microfabricated FDNMR. The trench is for optical fiber access.

e. *I-beam silicon*: It has the functions of r.f. excitation, sample container and FDNMR assembly.

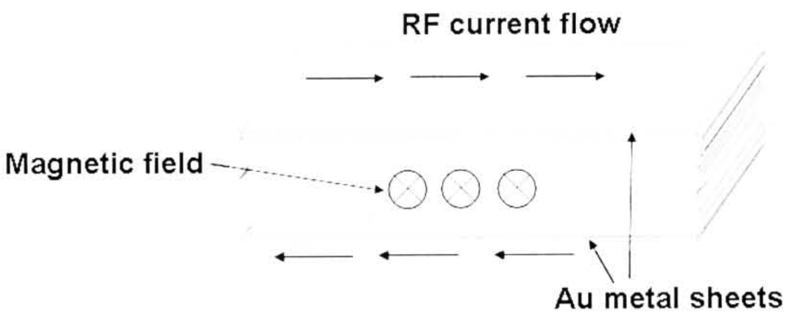

Figure 5. Concept drawing of I-beam. Au metal sheets on the side of silicon are for r.f. excitation.

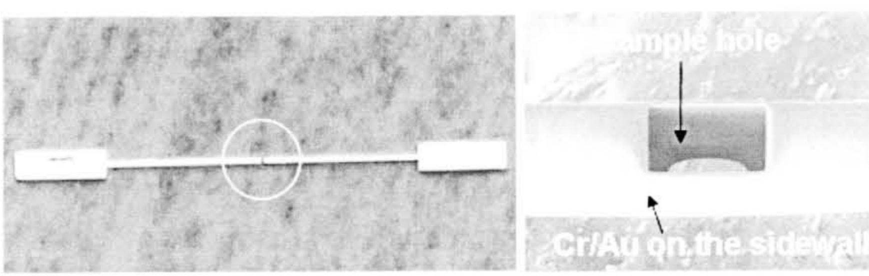

Figure 6. Microphotograph of "I-beam."

i. r.f. excitation: The most common excitation coil for NMR is the solenoid. Solenoids do not provide rigid structures for sample mounting and without significant modification do not allow for effective dissipation of heat during excitation. Heating of the mechanical oscillator causes the resonance frequency to drift during an experiment, resulting in decreased efficiency in driving. Therefore, we have constructed a new design which has imply consists of two parallel conductive sheets evaporated on a silicon "I-beam".

ii. Sample container: In the center of "I-beam", there a hole to allow placement of samples. Then, there is no concern of precise position of the sample.

iii. FDNMR assembly: One of challenge in the final assembly of the MEMS spectrometer involves achieving the precise alignment necessary for the various parts of the spectrometer to be put together. In addition, the RF excitation coil and the sample need to be fabricated and co-located within the spectrometer. The end of the I-bar has been used for precise alignment of FDNMR devices and "I-beam".

f. sample mold: In a liquid sample case, it is impossible to put it into the center hole in the "I-beam". The sample mold is used to capture the liquid sample using a parylene coating.

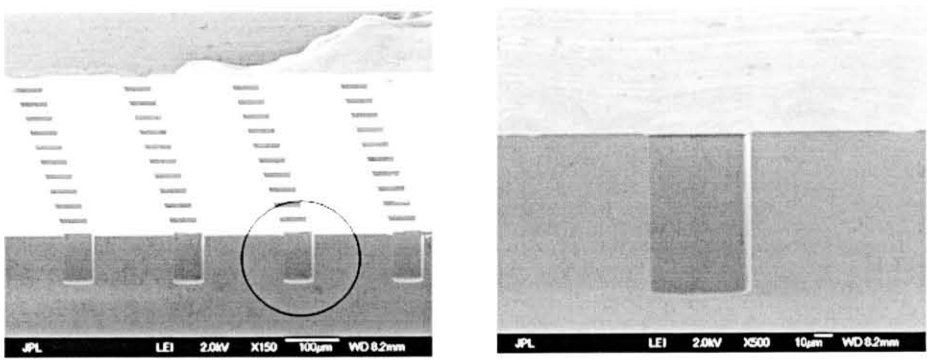

Figure 7. SEM image of a sample mold

g. assembled FDNMR device

We have successfully assembled the FDNMR devices as shown in Figure 8. We are trying to measure the resonance frequency of a resonator and hope to start to measure the FDNMR spectrometer soon.

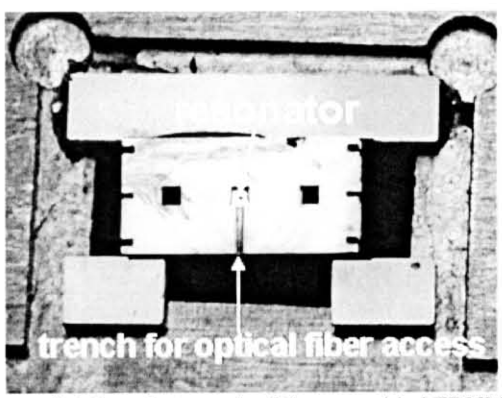

Figure 8. Microphotograph of the assembled FDNMR.

4. SUMMARY

We have designed the FDNMR spectrometer and successfully microfabricated FDNMR devices, I-beam, sample container, and so on. In addition, we successfully assembled the devices to build the FDNMR spectrometer.

Currently, the MEMS FDNMR spectrometer technology is at a lower maturity level. However, it promises to augment the NASA instrument "toolset" with an exciting new astrobiology capability. Ultimately, we hope to integrate the MEMS FDNMR spectrometer subsystem within a multi-instrument suite for in-situ planetary astrobiology exploration.

5. ACKNOWLEDGEMENT

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