Understanding Dissipation in Nanostring Resonant Sensors

CNF Project # 599-96

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Abstract

We have used optical drive and detection to study dissipation in silicon nitride beam resonators. We are able to tune resonant frequency as well as quality factor in a given nanostring resonator by several hundred percent by tuning the stress in real time, providing a unique look into the sources of dissipation for this class of nanoresonator. A record room temperature quality factor of 390,000 at 3.7 MHz has been attained in vacuum with a silicon nitride nanostring under tension. Nanostrings have also been resonated in viscous liquids including water and biological buffer. This is the first demonstration of the operation of truly nanoscale flexural resonators in viscous liquids.

Summary

Optical drive and detection have been used to actuate and detect the resonance of doubly-clamped nanostring resonators. These resonators have been made from both high and low-stress silicon nitride. Devices in a high stress state have been operated with quality factors at radio-frequencies in excess of $10^5$ MHz. In Figure 1, we show the resonance of a 75 µm long, 500 nm wide, 105 nm thick string, with a quality factor of 390,000 at 3.7 MHz [1]. The stress state can then be tuned for a device of a given size by bending of the resonator chip, as shown in the schematic in Figure 2. Using this chip-bending technique, we have been able to drastically tune beam stress, resulting in tuning of frequency by several hundred percent. Over this wide range of frequency tuning, the quality factor is also tuned by several hundred percent, as summarized in the plot in Figure 2 in which frequency is increased while dissipation is decreased for several devices of a given size (colored symbols), and frequency and dissipation are pushed towards the values for the as-fabricated high stress devices (black squares).

Future work on the source of this drastic quality factor tuning will include investigations into the influence of the nanostring clamping, which may be influenced by the addition of stress. Nanostring resonators have also been operated in gas, of potential utility for real-time chemical sensing, and viscous liquids (schematic shown in Figure 3) of potential utility for the sensing of biological molecules in real time. A 2 µm long, 160 nm wide, 125 nm thick metalized string was operated with a frequency of 145 MHz and quality factor of 400 in room air, and with a reduced frequency of 95 MHz and quality factor of 5 in water [2]. The resonance responses for this device are shown in Figure 3.

References


Figure 2

Chip bending allows stress tuning

Tensioning summary

Frequency (MHz)

Dissipation (1/Q)

Length varied

Tensioning

Figure 3

Optical drive and detection in liquid

Normalized resp. amp.

Air

Water

Q~400

Q~5

Frequency (MHz)
Resonant Micro- and Nanomechanical Biosensors

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Abstract

In this work we fabricate micro- and nanoscale devices, and use them to detect biological analytes from solution. Masses that bind to these devices can be weighed by monitoring the frequency response of the resonating devices. Careful functionalization of sensor surfaces gives them high specificity, allowing frequency shifts to be associated with analyte detection. These devices are promising candidates for compact test platforms in the detection of disease, harmful biological substances, and important biomarkers in a low-cost and rapid way.

Summary

Micro and nanoelectromechanical systems (MEMS and NEMS) are of interest for chemical and biological sensor applications due to their high sensitivities and small sizes [1]. These devices operate as miniaturized analogues of quartz crystal microbalances, exhibiting shifts in their resonant frequencies upon the addition of mass, such as bound biological species. Using nanoscale oscillators, sensitivity to added mass can be increased sufficiently for the detection of single bacteria [2], viruses [3] or molecules [4]. Resonant biosensors have demonstrated sensitivity to changes in mass as small as attograms [4]. The small scale of the devices encourages large scale arraying of sensors, building in appropriate redundancy and statistical samplings on a single chip. In addition, the high fill fraction of devices on a surface suggests that multiplexed detection of several different analytes from the same sample solution is possible.

Our devices are fabricated from low stress silicon nitride, with thicknesses on the order of 100 nm. Typical devices have lengths and widths on the order of hundreds of nanometers to a few microns. Variants of the standard cantilever beam shape, in addition to other interesting structures, are being investigated to improve sensitivity in the detection of dilute analytes. Resonant structures are fabricated by patterning the nitride device layer on a sacrificial layer of thermally oxidized silicon. Isotropic etching of the oxide releases the devices, making them free to oscillate. Then we functionalize our devices with a linking chemistry specific to the desired analyte. A schematic of the fabrication process is shown in Figure 1 for an immunospecific cantilever sensor.

We use an optical system to both excite and detect device resonance. Light modulated at the resonant frequency of the sensor is used to periodically heat the device and drive the oscillations. Optical interrogation of devices is performed using optical interference techniques. As the sensor is resonating, the intensity of reflected light changes due to the varying gap between the device and silicon wafer. This method allows the exploration of large arrays of devices as well as creative and nontraditional device structures that would be difficult or impossible to detect using other techniques.
For sensitive detection of biological entities, specificity is required, as resonant NEMS will respond to any adsorbed mass. We have worked to functionalize sensor surfaces using appropriate linking chemistries in order to present a surface of bound receptors like antibodies, for example, that are specific to a particular analyte. Reducing nonspecific binding of proteins to the nanomechanical biosensors is also an important consideration. With sufficient confidence in the specificity of the cantilever surface, in addition to appropriate controls, resonant frequency shifts can be attributed solely to adsorption and thus detection of the desired analyte. Current work is directed towards the detection of small (on the order of 10s of kDa) proteins important in the early detection of disease. The small mass of these analytes exacerbates nonspecific binding issues and requires further optimization of blocking chemistries in order to prevent false positive signals.

References


Vibrational Manipulation of Microspheres Using Optically Excited In-Plane Motion of NEMS Oscillators

CNF Project # 762-99

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Abstract

Optical excitation plays an important role in the actuation of higher flexural and torsional modes of nanoelectromechanical oscillators. We show that optical fields are extremely efficient for excitation, direct control and measurement of in-plane motion of cantilever-type nanomechanical oscillators. As a model system, 200 nm and 250 nm thick single crystal silicon cantilevers with dissimilar lengths and widths ranging from 6 to 12 µm and 500 nm to 1 µm, respectively, were fabricated using surface micromachining and dynamically analyzed using optical excitation and interferometric detection. Higher order modes of slender cantilevers, calculated using linear Euler-Bernoulli beam model, differed significantly from the measured values. Three dimensional finite element analysis incorporating shear, rotational inertia, cross-sectional deplanation and non-ideal boundary conditions due to the structural undercut, are shown to adequately describe the dynamics of the nanomechanical structures. The quality factor of a particular in-plane harmonic was consistently higher than the transverse mode. The increased dissipation of the out of plane mode was attributed to material and acoustic loss mechanisms. The in-plane mode was used to demonstrate vibrational detachment of sub-micron polystyrene spheres on the oscillator surface. In contrast, the out of plane motion, even in the strong non-linear impact regime, was insufficient for the removal of bound polystyrene spheres. Our results suggest that optical excitation of in-plane mechanical modes provide a unique mechanism for controlled removal of particles bound on the surface of nanomechanical oscillators [1].

Summary

Our cantilevers were fabricated from commercially available silicon-on-insulator (SOI) (100) wafers (SOITEC). The top structural silicon layer was 200 nm and 250 nm thick with a measured uniformity of less than 2%. Devices of varying length (l = 6-12 µm) and width (w = 500 nm-1 µm) were defined using optical projection lithography and etched with a CF₄ plasma. The exposed 1 µm thick sacrificial SiO₂ layer was removed using 49% hydrofluoric acid. Dynamic properties of the cantilevers were calculated and modeled for the geometry determined from optical and scanning electron microscopy. Rounding of the corners at the clamped and free end, due to optical proximity effects during lithographic definition of the beam, along with the undercut was incorporated into the three dimensional finite element model.

In order to identify the type of vibrational mode, the eigenfrequencies of the cantilever were first calculated and the corresponding eigenmodes were built using beam theory. The ratio of the two oscillatory modes (in-plane and out-of-plane) yields a dimensionless quantity $\phi = w/t$. For the fabricated devices of width 1 µm and thickness 200 nm, $\phi = 5$. For the devices with dimensions $l = 6.6$ µm, $w = 1$ µm and $t = 200$ nm, the first eigenfrequencies for the transverse and in-plane modes are shown in Figure 2a and 2b respectively. The measured ratio between the two modes $\phi_{measured} \sim 5.12$ was in good agreement with the Euler-Bernoulli theory. With this oscillator length, second and third transverse harmonics were also measured, however, higher in-plane vibrational modes were not detected. Additionally, excitations above the third out-of-plane harmonic were not observed. This is attributed to the dramatic contrast of the compliance between the two modes. Neglecting rounding of the corners at the clamped and free end, calculated compliance for the transverse and in-plane directions were $c_o = 0.855$ m/N and $c_i = 0.043$ m/N respectively. However, with fabricated devices of lengths exceeding 8.5 µm, we measured up to the second in-plane mode.
In the following treatment, as a model system, evaluation of employing in-plane oscillations to remove weakly bound polystyrene spheres was considered. First, polystyrene spheres with diameter ranging from 500 nm to 1 µm diameter were randomly dispersed on a chip containing a periodic array of a large number (> 10^6) of NEMS resonators. Oscillators containing single latex spheres were then identified and placed into a chamber that was evacuated to a pressure of 2 x 10^{-7} Torr. NEMS devices with single latex spheres were baseline measured using a low excitation signal. In this work, vibrational excitation and motion detection were accomplished using an optical setup comprised of an amplitude modulated 415 nm diode laser in conjunction with a HeNe interferrometric system (Figure 1). The incident HeNe laser power was 56.4 µW during the baseline measurements. The excitation source was then swept from 500 µV to 30 mV. In most cases, the excitation source had to be moved to one side of the clamped end (base of the cantilever) in order to introduce a thermal gradient in an effort to amplify the in-plane vibrations.

Figure 2 shows the in-plane first vibrational mode with the sphere and following the removal of the sphere by shaking. Here the oscillator had dimensions \( w = 601 \text{ nm}, l = 10.12 \mu \text{m} \) and \( t = 250 \text{ nm} \). Figures 3a and 3b show oblique angle scanning electron micrographs of the cantilever with a polystyrene sphere near the free end and directly following the removal of the bead respectively. Baseline measurements on a collection of oscillators with these dimensions had a natural out-of-plane harmonic of 3.16 ± 0.12 MHz. This was in excellent agreement with the results following the removal of the sphere.

References

Dynamic Characterization of Nanomechanical Oscillators

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Abstract
Dynamic detection of vibrational characteristics of nanoelectromechanical systems (NEMS) was investigated through direct coupling with a micromechanical probe. The nanomechanical structures were harmonically driven using piezoelectric transducers and the resulting out-of-plane excitations were monitored with a conventional atomic force microscope (AFM) probe. Intermittent contact imaging data show quantitative linear classical resonance behavior. Additionally, non-contact AFM interrogation revealed the initiation of interaction between the two oscillators, providing a qualitative description of the resonant response. The vibrational spectra measured through optical excitation and detection was in good agreement with the coupled NEMS-AFM system measurement results. The dynamic response of the coupled system was modeled through a combination of long range van der Waals and contact forces using the Derjaguin-Muller-Toporov model. These results collectively demonstrate that this is a viable method for detecting the dynamic behavior of nanoscale suspended mechanical structures [1].

Introduction
The desire to understand and control dynamical properties of micro and nanoelectromechanical systems [1–4] (MEMS and NEMS) has inspired great interest in a wide spectrum of applications, including scanning force microscopy, ultrasmall force detection, single charge detection, magnetometry, cavity quantum electrodynamics, chemical and biological sensing, parametric resonance, data storage, and mechanical mixers and filters. Dynamic detection of forced deflection or out-of-plane translational vibrations is generally accomplished by either optical interferometry or optical deflection techniques. A fundamental constraint in optical detection is encountered when the device dimensions approach the diffraction limit of the optical system. In this regime, for a diffraction limited laser spot size at the position sensitive detector and noise dominated by shot noise, reduction of the total laser power reflected from the surface of the nanomechanical device significantly degrades the signal to noise ratio. To circumvent these restrictions, a number of detection techniques have been implemented, including, electrostatic, magnetic, piezoelectric, optical pumping and mechanical actuation and detection utilizing a combination of scanning tunneling and electron microscopes, respectively. These methods of forced actuation generally require supplementary fabrication steps such as deposition of additional layers on the oscillator surface, which in turn alters the mass and hence the dynamical properties of the resonator.

Scanning probe microscopy represents an alternate approach to enhance and exploit the dynamical response of nanoscale systems through coupled mechanical interactions. Methods of measuring frequency response of large micromechanical structures using contact mode atomic force microscopy (AFM) have been reported. In these cases, the mass and stiffness of the structure under consideration were much higher than the AFM cantilever. With structures of reduced dimensions, the strongly interacting contact mode cantilever dominates the dissipative dynamics by dramatically modifying and broadening the vibrational properties of the probing NEMS device. Furthermore, due to this coupling, forces imposed during contact force imaging are sufficient to destroy small size and volume-to-surface ratio suspended structures.

Summary
In this article, we have studied the interaction between coupled MEMS and NEMS oscillators both experimentally and theoretically. This was based on an approach in which interactions between harmonically driven nanomechanical structures and an AFM probe in tapping and non-contact modes were used to illustrate the dynamics of the coupled system (Figure 1). For these experiments, suspended, surface micromachined single crystal, high frequency ($f_o$ 1 - 15 MHz) silicon NEMS cantilevers were fabricated...
and used in conjunction with commercially available AFM probes. The natural harmonic of the NEMS cantilever was much higher when in contrast with the probe with dimensions of length, \( l = 225 \mu m \), width, \( w = 30 \mu m \), and thickness \( t = 3 \mu m \) and a resonant frequency in the range of 50-100 kHz.

Low force, tapping mode imaging near the free end of a nanomechanical cantilever subject to a harmonic excitation revealed the frequency response of the NEMS device. The spectral response was most pronounced in the phase and amplitude images of the oscillating probe. Figure 2 displays amplitude of the probe dynamics in the vicinity of the cantilever natural harmonic. The Lorentzian fit reveals a resonant frequency of 5.01 MHz with a mechanical quality factor, \( Q = 3.9 \) for a single crystal silicon device with dimensions \( l = 7.92 \mu m \) and \( w = 526 \) nm.

In addition to topographic detection, the natural harmonic was measured using dynamic probing in intermittent and non-contact modes. In the former, the probe was positioned directly above the free end of the cantilever. The z-position of the probe was controlled by the extension and retraction cycle of the z-piezo. During the cyclic loading, in the fully retracted mode, the probe undergoes free oscillations. As the probe extends, it is lowered and brought in close proximity of the surface, the amplitude decays and finally jumps into contact with the surface. The point of contact marks a significant decrease in the vibrational amplitude of the probe (see Figure 3). The amplitude reaches a plateau throughout the range of the contact extension cycle. During the probe-cantilever retracting cycle, the vibrational amplitude remains approximately constant until the point when the probe breaks contact with the surface. The amplitude gradually increases and reaches a steady state value within the free vibration regime. Figure 4 shows the measured frequency spectra of the driven cantilever structure within the intermittent contact regime.

References


Figure 1: Illustration emphasizing the weakly coupled, non-contact and scanning modes of operation.

Figure 2: Tapping mode AFM scans across the piezo-driven cantilever between 3.5 - 6.5 MHz.

Figure 3: Measured extension-retraction cycle of the probe amplitude versus the z-piezo scanner position.

Figure 4: Measured vibrational amplitude (open circles) of the probe, near the vicinity of the point of contact.
Mechanics of Nanometer-Thick Suspended Carbon Materials

CNF Project # 789-99
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Abstract

We fabricated nanoelectromechanical systems (NEMS) from graphene sheets by mechanically exfoliating thin sheets [1, 2] over trenches in silicon dioxide substrates. The thinnest resonator consists of a single suspended layer of atoms and represents the ultimate limit of a two dimensional NEMS.

Background

In collaboration with the McEuen and Davis research groups, we continue to work on patterning, control and suspension of carbon nanotubes, but our major focus has been on suspended thin films of graphite, down to single atomic layers of graphene.

Figure 1: An optical micrograph of a suspended few-layer graphene sheet also measured to be 2 nm-thick by AFM.

Summary of Research

An optical micrograph of a suspended few-layer graphene sheet is shown in Figure 1. This particular membrane was measured to be 2 nm-thick by atomic force microscopy (AFM). Vibrations with fundamental resonant frequencies in the megahertz (MHz) range are actuated either optically via thermal expansion and contraction [3], or electrically by applying a radio frequency voltage relative to a doped silicon back-gate [4]. Motion is detected optically by laser interferometry. We detect the thermal motion of the resonators, and use the equipartition theorem to calibrate the amplitude of motion with the optical signal. AFM and spatially resolved Raman spectroscopy are used to determine the thickness of the suspended sheets [5].

Figure 2: A surface plot of the spring constant of a suspended graphene sheet vs. the location of the AFM tip.
Mechanical properties are measured using calibrated AFM probes [6] to measure static deflections in response to a known point force. We have made a detailed study of the mechanical properties of these resonators including resonance frequency, spring constant, built in tension, and quality factor. Figure 2 shows a series of measured values for the effective spring constant across the surface of a stack of suspended graphene sheets which are shown in an AFM image in Figure 3 with a corresponding grid. On layered graphene sheets of thicknesses between 2 nm and 8 nm, we measured spring constants ranging from 1 to 5 N/m. Our data is fit to a model for doubly clamped beams under tension by plotting the spring constant, as measured in the center of the device, vs. $w(t/L)^3$ for eight different suspended graphite sheets. As shown in Figure 4, we can use this fit to extract a Young’s modulus of 0.4 TPa, compared to 1 TPa for bulk graphite along the basal plane, and tensions on the order of 10-7 N [7]. The unusually small mass, electrically active material and reasonable dynamic range indicate that graphite resonators would make excellent mass and charge sensors.

References


Figure 3: An AFM amplitude micrograph of the suspended sheet and overlay grid corresponding to Figure 2. The vertical ‘stripe’ in the picture is the trench etched into the silicon dioxide.

Figure 4: A plot of the spring constant, as measured in the center of the device, vs. $w(t/L)^3$ eight different suspended graphite sheets. The dashed line is the fit to all where as the solid line is the fit for 7/8ths of the data.
An Electronic Nonvolatile Memory Device Based On Electrostatic Deflection of a Bistable Mechanical Beam

CNF Project # 804-99
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Abstract
The speed of memory structures is considerably slower than that of logic thus permitting a variety of approaches to achieving and using bistability. We used the bistability of a doubly clamped beam to attain two states that can potentially be used in the implementation of a nonvolatile memory [1]. The device is designed such that a bistable beam acts as the gate of an air gap transistor. In theory, in scaled structures, the threshold voltage shift corresponding to a gate deflection of nanometer dimensions provides sufficient change to allow sensing of the state of the nonvolatile memory cell.

Summary of Research
The bistable mechanism has found numerous applications in the microelectromechanical systems (MEMS) field related to mechanical switches and valves. Experiments by B. Halg [2] have demonstrated that it is also conceivable to use mechanical bistability in a nonvolatile memory cell. Similar work [3, 4] has shown simulations of the electrical characteristics of a metal oxide semiconductor field effect transistor (MOSFET) with a buckled floating gate. We have refined these concepts further and put them into practice by designing and fabricating a novel structure suitable for the integration of an FET (Figure 1). In addition, the process developed for this device is completely complementary metal oxide semiconductor (CMOS) compatible.

The write/erase mechanism of this memory device operates on the principle of electrostatic attraction. The plate is incorporated in the device to help perform the write function. The upper plate is nearly four times thicker than the gate thus making it highly stiff by comparison. This establishes the gate as the only deformable element of the device. Therefore, in order to obtain the “down” state (Figure 2), a large bias is applied between the gate...
and the source/drain, thus attracting the gate towards the channel. Upon removal of the voltage, the gate remains in the “down” position due to it being energetically favorable as a result of the bistable mechanism. On the other hand, in order to obtain the “up” state (Figure 3), a large bias is applied between the upper plate and the gate so that the gate is pulled up to the other stable position. The read function is executed using small biases between the gate and the substrate. In read mode, the device operates like a MOSFET. The read voltages must be lower than the write voltages in order to prevent any additional deflection of the gate. The device’s electrical characteristics substantially change as the gate is moved between the two stable states. The “down” state corresponds to a lower threshold voltage and sub-threshold slope, whereas the “up” state is described by a higher threshold voltage and sub-threshold slope.

Our device features a bistable polysilicon beam sandwiched between two silicon dioxide layers as seen in Figure 1. The bistable beam is obtained by patterning and releasing a compressively stressed thin film with sufficient internal stress to cause buckling upon release. This enables the bistable mechanism; the beam can be deflected either up or down, thus creating two stable states. The middle polysilicon layer is deposited using the low pressure chemical vapor deposition (LPCVD) furnace at 620°C for 12 minutes which results in a film thickness of 80 nm. In addition to the beam used for the gate, another beam or plate is suspended above the gate. The plate is also comprised of LPCVD polysilicon and has a thickness of 330 nm. The two silicon dioxide layers are both 1.1 µm thick and are made of low temperature oxidation (LTO). These dual LTO films serve not only as the sacrificial release layers, but also act as the anchors for the suspended beams. Furthermore, they also provide electrical isolation between the plate, beam, and substrate. After depositing a stack of the previously stated materials, a top down approach is taken to fabricate the devices. A bi-layer resist liftoff process is performed to leave 40 nm thick Cr features. Cr serves as a robust etch mask for the subsequent plasma etching. The ICP etcher was chosen for its enhanced etch rates and its ability to produce higher aspect ratio features than the standard parallel plate RIE systems. The stack was etched in CF₄ plasma and the etching was stopped in the bottom LTO layer. Etching was then continued in the parallel plate RIE system with CHF₃/O₂ to provide higher selectivity to the silicon substrate. In order to open up a via to the gate electrode a thick resist was used to protect the entire structure. A window was then opened in the resist so that the ICP etcher could be used to etch partially through the stack and stop in the top LTO layer. The resist was then stripped and the structure was dipped in BOE to complete the etching of the via while also removing the dual sacrificial oxide layers. Upon release, the beams were equally likely to buckle to the up or down state due to the highly symmetric design of the structure.

The design for this bistable mechanical memory device permits straightforward fabrication, thus making it possible to scale this structure down further. Having both the beam and plate defined in the same etch allows for a self-aligned process. In addition, the FET channel runs perpendicular to the beam-plate structure, thus permitting self-aligned implants. By incorporating novel and more compliant materials in this structure, device performance can be further improved, making this a highly competitive technology.

References
Mechanical Devices

Ultrasensitive, Magnet-Tipped Cantilevers for Magnetic Resonance Force Microscopy

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Abstract
Magnetic resonance force microscopy (MRFM) is a developing technology in the family of force microscopy techniques. MRFM detects magnetic resonance as a force on a magnet-tipped microcantilever facilitating three-dimensional, chemically specific subsurface imaging at the nanoscale [1]. If sufficiently high sensitivities can be reached, this technique could achieve atomic scale magnetic resonance imaging, and could be used, for example, to read out the structure of large biomolecules or to study buried semiconductor interfaces. An essential step in achieving the required sensitivity is the development of high sensitivity cantilevers with nanoscale magnetic tips. Our work at the CNF has focused on creating 50-200 nm wide cobalt magnets which extend from the tips of 5 µm wide silicon cantilevers.

Summary
The purpose for creating overhanging nanoscale magnets is to maximize the force exerted on the cantilever by each magnetic spin, while minimizing noise in the force signal that arises from non-contact frictional forces between the cantilever and the sample. To achieve single-spin sensitivity, the front of the magnet must be within a few nanometers of the sample. Work by our group [2] has found that, within tens of nanometers of sample surfaces, metal has less friction than silicon, and that narrow cantilever tips have less friction than wider ones. Thus our cantilever design has the magnet extending past the end of the silicon cantilever, and the very tip of the silicon cantilever is narrowed from 5 µm to 1. Figure 1 is of a 50 x 50 x 1000 nm overhanging Co magnet on a test structure. The dotted line outlines the location of the cantilever edges in the final product. Figure 2 shows the entire cantilever and base. The octagonal area on the cantilever is the laser pad for our interferometer based motion detection system.

The fabrication process starts with <111> oriented silicon-on-insulator wafers. Electron-beam lithography with the JEOL-9300 is used to define the magnets, which are created through thermal deposition of cobalt and subsequent liftoff. A thin layer of oxide is deposited using the GSI PECVD to protect the magnets in subsequent processing. Next, the electron-beam lithography is used to define rectangular etch pits adjacent to the magnets. Plasma etching is used to etch these pits through the device layer of the wafer. The wafer is then etched in heated KOH. The design of the etch pits, the <111> orientation of the silicon, and the anisotropy of the KOH etch combine to undercut the silicon below a portion of the magnets, leaving the magnets extending...
The work in the past year has focused on process integration. After exhaustive testing, we suspect that cobalt silicide formation is causing the degradation of the magnets during the Unaxis backside etch step. Our current work is in modifying the backside etch process to minimize heating of the magnets, and seeking ways of reducing or eliminating the PECVD oxide layers.

**References**


approximately 200 nm from the post-etch, etch pit edge [3]. The cantilever body is defined with the GCA Autostep, and etched.

A second, thicker layer of PECVD oxide is deposited to protect the devices during backside processing. The backside of the wafer is patterned using the EV-620, and etched through to the buried oxide layer using the Unaxis 770. The cantilevers are released in buffered hydrofluoric acid etch, followed by critical point drying. Non-overhanging magnets can also be fabricated using this procedure, by eliminating the KOH etch step. Figure 3 shows a 200 x 600 x 1500 nm non-overhanging magnet on the end of a cantilever. With this wafer the backside silicon etch was not completed, leaving the undesired material underneath the cantilever.

Figure 2: View showing entire cantilever and portion of base.

Figure 3: Close-up of cantilever tip with non-overhanging, 600 nm wide Co magnet.
Magnetic Resonance Force Microscopy
Cantilever Detection by Quantum Tunneling

CNF Project # 863-00
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Abstract
Magnetic resonance force microscopy is a novel characterization technique, with promising applications to nondestructive three-dimensional sample imaging. Magnetic resonance is detected as force on a magnet-tipped cantilever, with potential for atomic scale magnetic resonance imaging of biomolecules or semiconductors [1]. To achieve such a lofty characterization goal, high-$Q$ nanomechanical magnet-tipped cantilevers are required. Here, we present a fabrication project in progress at the CNF that aims to appease this need; the integration of tunnel-based displacement detectors into the MRFM cantilevers.

Summary of Work
Magnetic resonance force microscopy (MRFM) aims to interrogate nuclear spins locally by setting the Larmor spin resonant condition with high spatial selectivity [1]. This is accomplished by a nanomagnet on the tip of an RF nanoscale singly-clamped cantilever. In an attempt to eliminate coupling between the cantilever and detection interferometer, we are considering a tunnel-based displacement detector. The tunnel sensors themselves consist of a break junction [2] between the beam and a fixed electrode; as the beam oscillates, the junction separation changes, and the tunneling current through the junction varies measureably. Calculations have shown that the measurement sensitivity is approximately $10^{-13} \text{m}/\sqrt{\text{Hz}}$, well within that required for MRFM. More importantly, this technique is viable around ambient temperatures of 300 K, while alternative methods (RF-SET’s, etc.) require cooling. The tunnel sensors are patterned with the JEOL JBX-9300 dedicated electron-beam lithography system, with a feature size of 30 nm. Optical lithography is used to define the interconnects to the probe station. RIE is then used to define the cantilevers and junction underetch.

To date, we have generated a fabrication protocol for tunnel sensors in doubly clamped cantilevers. We are currently characterizing the doubly-clamped beams with the magnetomotive actuation technique. Upon completion of the characterization, we will integrate the tunnel-based displacement detection mechanism with the nanoscale tip magnets (in a singly-clamped beam) to create a new generation of MRFM cantilevers.

References
Figure 1: Here, we see an array of fabricated tunnel sensors.

Figure 2: Up close, one can see the separation between the tunnel sensor and fixed electrode.
AFM Tip Processing on Hinged Cantilevers

CNF Project # 883-00

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Abstract

A method for atomic force microscopy (AFM) tip preparation is described that is amenable for integration onto a hinged cantilever, thereby achieving an exquisitely high-sensitive probe. Undercutting an etch mask with successive reactive ion etching, results in a quasi-pyramidal, high-aspect-ratio tip. Processing steps will be discussed, scanning electron microscopy (SEM) images will be shown, and a short narrative of how the tips are integrated onto the cantilevers will be described, including future directions of the work.

Summary

Few modern instruments can lay claim to the level of success enjoyed by the atomic force microscope (AFM). The ability to map surface topography with ~ nm resolution finds usefulness in almost every branch of science.

The original AFM employed a scanning tunneling microscope (STM) to monitor the motion of a diamond-tipped stylus supported on a gold foil cantilever [1].

Today’s AFM typically employs a silicon micromachined cantilever with a sharp tip on one side, and a reflective mirror surface on the other [2, 3]. When the tip interacts with the test surface, its deflection is measured by reflecting a laser beam off the back side of the cantilever onto a 4-segment position-sensitive photodetector (PSPD). Whereas the top and bottom segments of the detector indicate height variations in the position of the tip, the left and right segments can be used to indicate the amount of friction between the tip and sample surface [4]. However, because the same cantilever is used in the vertical bending mode (for topographical imaging) and the lateral bending mode (for frictional force imaging) these motions are inextricably coupled with mechanical crosstalk resulting.

Chui [5] was arguably the first to appreciate decoupling of the lateral and vertical sensitivities in an AFM cantilever. Later, Kageshima [6] was able to mill a trench into a commercial AFM cantilever with a focused ion beam (FIB), thereby enhancing both the lateral and vertical sensitivity. Taking both of these concepts a step further, Beyder [7] was able to process silicon cantilevers with orthogonal (i.e. gimbaled) Si₃N₄ hinges of arbitrary thickness, thereby decoupling vertical and lateral sensitivities altogether. This allowed the geometry and stiffness to be separately optimized for each sensing direction. With arbitrary thickness hinges, Beyder is able to achieve exquisitely sensitive levers for the analysis of delicate soft-matter samples. Figure 1 exemplifies a dual-axis gimbaled lever with two tips (courtesy of Beyder).
In all of these cantilever arrangements, the tip that interacts with the test surface greatly influences the quality of the AFM image. Whereas the radius of curvature of the tip determines, in part, the imaging resolution of the cantilever, the shape of the tip can be more or less susceptible to image aliasing and artifacts that seriously degrade the final image [8].

At the CNF, silicon AFM tips are defined by a sequence of three reactive ion etch (RIE) processes: (1) isotropic etch to undercut the etch mask (O-Release), (2) anisotropic etch to lengthen the underlying pillar that results (O-Trench), and (3) a second isotropic etch to sharpen the pillar into a final quasi-pyramidal tip (O-Release).

A ~ 5 µm diameter circular etch mask comprising ~ 1.3 µm of S1813 (Shipley) photoresist, 30 nm of LPCVD nitride (low-stress, 800°C, 11 min) and 135 nm of thermal oxide (wet, no-HCL, 900°C, 65 min) is prepared.

For undercutting the etch mask, we utilize the default O-Release recipe in the Unaxis 770 ICP (inductively coupled plasma) tool. The O-Release program uses four etch steps of Ar (50 sccms) with increasing flowrates of SF₆ (20, 40, 60 and 70 sccms, respectively) at 850 watts of RF power. The duration of each of the four etch steps is 2, 2, 2 and 60 sec respectively. The resulting undercut mask is shown in Figure 2 with an underlying, partially-obscured pillar supporting the mask. On top of the mask, the resist is deformed into a 4-leaf clover-like formation.

To lengthen the pillar, we use the default O-Trench recipe on the Unaxis ICP tool. Each loop of the O-Trench program consists of a first polymer deposition step (70 sccms of C₄F₈, 2 sccms of SF₆, and 40 sccms of Ar, 5 sec at 850 watts RF-ICP) followed by a first etch step (2 sccms of C₄F₈, 70 sccms of SF₆ and 40 sccms of Ar, 2 sec at 850 watts RF-ICP) and a second etch step (2 sccms of C₄F₈, 100 sccms of SF₆ and 40 sccms of Ar, 5 sec at 850 watts RF-ICP). Five loops of the O-Trench recipe results in the pedestal/cap structure of Figure 3.

A final O-Release etch for 60 sec (in the last etch step) pinches-off the cap, and transforms the underlying pillar into a quasi-pyramidal tip, as shown in Figure 4.

Implementing one or more of these silicon AFM tips onto a single-axis or double-axis cantilever is done by lithographically defining the arms and hinged regions of the cantilever(s) after tip preparation.

Future work may include polarimetric encoding of the reflected optical signals from multiple, reflective regions on the back of a multi-axis, multi-tipped cantilever. Such encoding practices would allow a considerable increase in information content conveyed from the test surface.

The results presented in this work are due in part to the knowledge, expertise and superlative support provided by the CNF staff. This work was supported by the NIH and NSF.

References
Functionalized Micromechanical Resonators with High Quality Factors

CNF Project # 891-00
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Abstract

The dissipation of mechanical energy in 250-nm-thick, megahertz (MHz)-range silicon resonators is found to be strongly dependent on the chemical nature of the surface. As a result, sub-monolayer changes in the termination of 250-nm-thick resonators lead to significant changes in quality factor. The chemical origins of this effect are under investigation. Strategies for the formation of arbitrarily functionalized, high-\(Q\) resonators have been demonstrated.

Summary

The development of stable, high performance micro-and nanomechanical resonators would enable advances in many fields, including chemical and biological sensing. In these applications, an adsorbed mass leads to a small shift in resonant frequency. The sensitivity of resonance-based detectors depends crucially on the resonator’s stability and quality factor, \(Q\). (The \(Q\) of a resonator is proportional to the number of oscillations the resonator will undergo after an impulsive excitation. As a result, \(Q\) is inversely proportional to the rate of mechanical energy dissipation.) Therein lies the problem. Although advances in microfabrication have made the production of high frequency resonators almost routine, a general trend of decreasing quality factor with decreasing resonator size has been observed.

We have shown that mechanical energy dissipation in MHz-range, micromechanical resonators is often dominated by the chemical state of the surface. By changing a single monolayer of molecules on the surface of a 250-nm-thick silicon resonator—less than 0.07% of the total mass—the quality factor of the resonator can be improved dramatically [1]. In contrast, the standard commercial coating, a thin layer of silicon oxide, dissipates at least 75% of the mechanical energy in similarly sized resonators. This result shows that chemical control down to the monolayer level will be necessary for the production of high-performance nanomechanical sensors. We are working to understand and control this effect.

Figure 1: MHz-range resonator fabricated from Si(111) wafer and suspended by 450-nm-wide silicon wires.

In recent experiments [2], we have shown that silicon oxidation does not inherently lead to increased mechanical energy dissipation. When silicon surfaces are oxidized in a controlled, strain-free manner—such as by the introduction of one-half monolayer of Si-O-R surface groups—increased dissipation is not observed. This
result is important, because it opens the door to a much broader range of functionalization chemistries for high-performance sensor applications.

In related work, we have developed a robust platform chemistry for the production of high quality resonators with essentially arbitrary organic functionality. This process uses Grignard reagents to produce a partially alkene-coated surface followed by a catalyzed olefin methathesis reaction that attaches the desired functionality to the surface alkenes. This strategy is sketched in the accompanying figure. As proof of concept, we have produced ester-functionalized resonators with quality factors near those of our best performing surface coatings.

References


Figure 2, above: The effect of surface chemistry on the quality factors of 5 MHz resonators.

Figure 3, below: Strategy for the production of high-Q functionalized resonators.
Electromechanical Resonators from Graphene Sheets

CNF Project # 900-00
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Abstract
Nanoelectromechanical systems were fabricated from single and multilayer graphene sheets by mechanically exfoliating thin sheets from graphite over trenches in SiO₂. Vibrations with fundamental resonant frequencies in the MHz range are actuated either optically or electrically and detected optically by interferometry. We extract a Young’s modulus of 1 TPa, and find that most suspended sheets are under tension. The quality factors of the suspended graphene sheets are in the range of 20-850 and show no dependence on the thickness. The thinnest resonator consists of a single suspended layer of atoms and represents the ultimate limit of two dimensional nanoelectromechanical systems.

Research Summary
The miniaturization of electromechanical devices promises to be revolutionary in the coming decades as the miniaturization of electronic devices was in the previous ones. Devices ranging from nanoscale resonators, switches, and valves have applications in tasks as diverse as information processing, molecular manipulation, and sensing. The prototypical nanoelectromechanical system (NEMS) is a nanoscale resonator, a beam of material that vibrates in response to an applied external force. The ultimate limit would be a resonator one atom thick, but this puts severe constraints on the material. It should be robust, stiff, and stable as a single layer of atoms.

Graphite consists of stacked layers of graphene sheets separated by 0.3 nm and held together by weak van der Waals forces. It has extremely high strength, stiffness, and thermal conductivity along the basal plane. In addition, graphite can be exfoliated onto an insulating substrate, producing micron-sized graphene sheets with thicknesses down to a single atomic layer. Thus far, research on these thin graphene sheets has focused primarily on their electronic properties. We demonstrate a method of suspending single and multilayer graphene sheets over trenches and show such sheets can be mechanically actuated. This work also makes a detailed study of the mechanical properties of these graphene resonators including resonance frequency, spring constant, built in tension, and quality factor.

Suspended graphene sheets are fabricated using a mechanical peeling process where the graphene sheets are exfoliated off of bulk graphite predefined trenches etched into a silicon oxide surface. Figure 1 shows that these sheets are optically visible under a microscope. The result is a micron-scale doubly clamped beam or cantilever clamped to the silicon oxide surface via van der Waals attraction (Figure 2). Some devices have pre-patterned gold electrodes between the trenches to make electrical contact [1]. Using this method, we have fabricated suspended sheets with thicknesses varying from a single atomic layer to sheets 75 nm thick.

The resonators are actuated using either electrical (Figure 3) or optical modulation. In the case of electrical modulation, a time-varying radio frequency (RF) voltage is applied to the graphene sheet resulting in an electrostatic force...
between the suspended graphene sheet and the substrate. For optical actuation, the intensity of a diode laser focused on the sheet is modulated at the resonant frequency of the graphene sheet, causing a periodic contraction/expansion of the layer that leads to motion. In both cases, the motion is detected by monitoring the reflected light intensity from a second laser using a fast photodiode. Figure 4 shows the measured amplitude versus frequency of a single atomic layer graphene resonator, with a measured resonance frequency of 70.5 MHz and $Q = 78$. The resonance frequencies of the fundamental modes of the fabricated sheets vary from 1 MHz to 166 MHz with quality factors, $Q$ of 20-850.

By comparing the resonance frequency of the suspended sheets to the measured size dimensions, we can deduce a Young’s Modulus of 1 TPa using continuum beam mechanics. However, the frequencies of thinner resonators (under 7 nm) show more scatter with the majority having resonant frequencies significantly higher than predicted by bending alone. A likely explanation for this is that many of the resonators are under tension, which increases the resonance frequency. The tension likely results from the fabrication process, where the friction between the graphite and the oxide surface during mechanical exfoliation stretches the graphene sheets across the trench.

An important measure of any resonator is the normalized width of the resonance peak characterized by the quality factor $Q = f_r/\Delta f$. A high $Q$ is essential for most applications, as it increases the sensitivity of the resonator to external perturbation. We measure $Q$ in the graphene sheets under vacuum of 20 to 850. This contrasts with diamond NEMS (2000-4000), silicon nitride NEMS (up to 400,000 now), and carbon nanotubes (50-100). Preliminary studies on a 20 nm thick resonator found a dramatic increase in $Q$ with decreasing temperature ($Q = 100$ at 300 K to $Q = 1800$ at 50 K). This suggests that high $Q$ operation of graphene resonators should be possible at low temperatures.

The high Young’s modulus, extremely low mass, and large surface area make these resonators ideally suited for use as mass, force, and charge sensors. For a 5 nm suspended sheet where we can detect the thermal vibration, we infer that we should be capable of measurements with a mass sensitivity of 7 zg, a force sensitivity of .9 fN/ Hz$^{1/2}$, and a charge sensitivity of $8 \times 10^{-4}$ e/Hz$^{1/2}$. These values are competitive with state of the art silicon NEMS at room temperature, and could get much better at lower temperature with the onset of higher $Q$.

However, the application of graphene NEMS extends far beyond just mechanical resonators. This robust, conducting, membrane can act as a nanoscale supporting structure or atomically thin membrane separating two disparate environments.

References
**Temperature Measurement of Boiling Flow in Microchannels with Reentrant Cavities**

**CNF Project # 1097-03, 1180-03**

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### Abstract

Experiments were conducted to investigate flow boiling in 200 µm x 253 µm parallel microchannels with structured reentrant cavities. Flow morphologies and local temperature measurements were obtained and studied.

### Introduction

Boiling enhancement technique has been a topic of great interest over the last fifty years [1], and is a rich field of current study. Of particular engineering success are special heat transfer surfaces encompassing re-entrant cavities that promote bubble ebullitions [2, 3]. Inspired by the rapid development of flow boiling in micro domains, research endeavors aimed at exploiting recent advances in microfabrication technologies to form structured surfaces that enhance heat transfer for flow boiling are currently underway [4-6]. The current investigation presents an examination of flow boiling through 223 µm hydraulic diameter microchannels with re-entrant type cavities. Flow boiling patterns and local temperature measurements are presented and discussed.

### Device Overview and Experiment Procedures

The silicon microchannel device consists of five parallel 10,000 µm long, 200 µm wide and 253 µm deep microchannels spaced 200 µm apart. Each sidewall encompasses an array of 100 re-entrant cavities spaced 100 µm apart, as shown in Figure 1. An acute angle connects the 7.5 µm mouth to the 25 µm inside diameter reentrant body. In order to minimize ambient heat losses, an air gap is formed on the two ends of the side walls, and an inlet and exit plenum are etched on the thin silicon substrate (~ 150 µm). A 20 µm wide and 400 µm long orifice is installed at the entrance of each channel inlet to suppress flow instabilities. The microchannel, re-entrant cavities, and orifices were all fabricated from deep reactive ion etch (DRIE) process. At the top of the device, a Pyrex® cover seals the device from the top and allows flow visualization.

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**Figure 1:** SEM image of reentrant cavities.

**Figure 2:** CAD models of the heater and the thermistors on the back side of the micro device.
Figure 2 depicts a CAD model of the backside of the device. Three aluminum thermistors 10 μm wide and 300 μm long are located 3,400 μm, 6,700 μm and 10,000 μm downstream of the channel inlet together with electrical connecting vias. On top of the thermistors layer, a 1 μm silicon oxide layer is deposited for electrical insulation. An aluminum heater is then formed on top of the oxide layer to deliver the heating power and also serves as a thermistor to measure the average temperature of the entire microchannel area.

The microchannel device is mounted on the fluidic packaging module through o-rings to ensure a complete leak-free system. The fluidic packaging delivers the working fluid (water) and access to the pressure transducers. The heater, which is fabricated on the device backside, is wired (through electric pads) to the power supply. The thermistors are also connected to a data acquisition system. The boiling process in the microchannels is recorded by a high-speed camera mounted over a microscope. During the experiment, voltage was applied in 0.5 volt increments to the test section heater, and the data for the heater and the thermistors were recorded once steady thermal-hydraulic state was reached, at which water flow rate, heat input, and temperature data remained constant. The procedure was repeated for different flow rates.

Results and Discussion

Four prime flow patterns were visualized in the reentrant cavity microchannels prior to the critical heat flux (CHF) conditions: single-phase, bubbly (Figure 3), slug, and annular flow patterns. The extent of the bubbly flow pattern was shown to be dependent on the mass velocity and heat flux. The slug flow was characterized by a large vapor core that occupied the entire microchannel cross-section and oscillated rapidly over several hydraulic diameters. Downstream, a more stable annular flow was developed, which was characterized by a vapor core engulfed by a continuous liquid sublayer attached to the microchannel walls. As the mass quality increased, the liquid layers diminished until dry spots at the exit region were detected indicating the arrival of the critical heat flux conditions.

Figure 4 shows $T_2$ as a function of the effective heat flux ($q''_{\text{eff}}$) for four different mass velocities. As expected, at low heat fluxes, single-phase liquid flow prevails, which is depicted by the linear temperature increase with increasing surface heat flux. When the heat flux is further increased, a significant shift in the $T - q''_{\text{eff}}$ slope is apparent marking the onset of nucleate boiling. Flow visualization reveals small bubbles emerging from the reentrant cavities near the channel exit. As the heat flux was further increased above a certain value, the average surface temperature abruptly surged with a meager rise of heat flux, indicating the emergence of critical heat flux condition. The CHF was verified by flow visualizations of dry spots at the channel exit region.

References

Directional Microphone Array Fabrication

CNF Project # 1116-03
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(click on: Faculty Research, then Microacoustic Sensors Laboratory.)

Abstract
The goal of this project is to fabricate prototypes of novel silicon microphone arrays. The microphone arrays consist of two differential microphones and one omni-directional microphone on each die. The miniature directional microphone technology is being developed for applications in hearing aids.

Summary of Results
Several new microphone designs and fabrication processes were developed and refined this past year. In collaboration with Prof. Levent Degertekin at Georgia Tech and Prof. Douglas Jones at the University of Illinois Champaign-Urbana, we have developed a prototype packaging scheme for our microphones so that our directional microphone array is small enough to fit on a hearing aid, as shown in Figure 1. The microphone array consists of two differential microphones and a nondirectional microphone located on the same silicon chip. The microphone package contains optoelectronic components to produce electronic outputs from the three microphones.

The microphone diaphragms we are developing are designed to respond to spatial gradients in the incident sound pressure by rotating about hinge supports as shown in Figure 2. An optical detection scheme has been developed to convert the sound-induced motion of the diaphragm into an electronic signal as shown in the figure. This consists of a semiconductor light source (VCSEL), an optical grating on the diaphragm, and photodetectors. We have shown that this optical scheme, along with the novel diaphragm design, has resulted in a high sensitivity and low-noise directional microphone.

The internal noise of miniature directional microphones is a significant design issue because the acoustic sensitivity of the device reduces with the diaphragm size. Figure 3 shows measured sound input-referred thermal noise of the microphone along with the results of an analytical prediction. This small directional microphone, with diaphragm size of 1 mm by 2 mm, has a thermal noise floor of 36 dBA, which is much lower than can be achieved in a directional microphone using conventional technology with a diaphragm of this size.

Figure 1: The prototype microphone package is small enough to fit in a hearing aid.
Figure 2: Schematic showing the optical sensing scheme to detect the sound-induced motion of the diaphragm.

Figure 3: Measured sound input pressure-referred thermal noise of our 2 mm by 1 mm directional microphone diaphragm.
Fabrication of Thin-Walled and High-Aspect-Ratio Nanofluidic Channels

CNF Project # 1176-03
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Abstract

Nanofluidic structures promise to solve the sample preparation problem in various spectroscopy experiments where the thickness of the sample is usually constrained by the penetration depth of the optical probe. We have designed and successfully fabricated a nanofluidic cell which was used in a two-dimensional infrared (2D-IR) spectroscopy experiment to study the nature of the OH bond in water [1, 2]. The fabricated structure employs a system of access holes and channels used for sample delivery and active thickness control.

Summary of Research

The building blocks of the nanofluidic cell are two free standing membranes of low stress silicon nitride back-etched on separate wafers using potassium hydroxide (KOH). A silicon oxide spacer deposited on one wafer was used to define a gap between the silicon nitride membranes when the matching pieces of the structure are bonded together. The etching of the silicon oxide layer to create the actual gap is done with 6:1 buffered oxide etch (BOE). In the first generation cell, the thicknesses of the silicon nitride windows were 800 nm, and the cell was filled from side channels, then sealed, trapping the liquid inside. In such a passive approach, the instability of the thin silicon nitride membranes leads to a poorly defined sample thickness.

In recent designs, input and output access holes were etched into the surface of the structure as shown in Figure 1, then connected to an external pump system. The active control of the gap thickness was realized using the transmitted intensity of an IR beam through the cell as feedback signal. In addition, a 70 µm deep KOH etched channels were used to connect the narrow gap to the access holes therefore restricting the high flow resistance region to the sample area. Lastly, a hydrophilic surface is created by high temperature deposition (LPCVD) of ~ 10 nm silicon oxide [3] in order to enhance the filling of the cell. An assembled cell filled with water is shown in Figure 2. The variation in sample thickness is most apparent from the interference rings.

References

Figure 1: Schematic of nanofluidic device for nonlinear spectroscopy on thin sample liquids.

Figure 2: Nanofluidic cell filled with water. Edge thickness is ~1500 nm, center thickness is ~400 nm, window size 1 x 1 mm.
Fabrication of Gecko-Inspired Spatula Tips

CNF Project # 1201-04
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Abstract

Insects such as flies and small animals such as geckos employ micro and nano-scale fibers on their feet to achieve excellent attachment capability. However, these animals must also possess the ability to quickly switch between good adhesion and easy detachment, which is essential for locomotion. It is thought that the geometry of the foot fibers (tilted fibers) and that of the fiber tips (asymmetric arrangement) play a central role in achieving such a switching mechanism. In this project, we aim to fabricate a gecko-inspired surface with asymmetric tips, similar to an inverted “L”. Such surfaces are expected to possess interesting anisotropic adhesion and frictional properties of technological interest.

Summary

Following the recent work [1] in demonstrating that the micro and nano-scale architecture of the gecko foot fibers is responsible for the animal’s excellent attachment capability, there have been several investigations to describe the mechanics of insect attachment as well as to create surface engineering strategies to mimic it [2-6]. Some of the successful strategies so far include arrays of fibers with mushroom shaped tips [2-3], film terminated fibrillar arrays [4], surfaces with wrinkled patterns [5], arrays of aligned carbon nanotubes [6], etc. A key feature of gecko and other insect attachment is the ability to quickly switch between strong adhesion and easy detachment, which is essential in locomotion. It is thought that the fiber geometry and the fiber tip geometry play an important role in such a switching mechanism.

One of the goals of this project is to investigate the mechanics of individual mechanisms which contribute to the switching mechanism and develop surface engineering strategies of technological interest.

In order to fabricate polydimethylsiloxane (PDMS) fiber tips with asymmetric tips, we developed a process in which a silicon mold of the corresponding shape is fabricated. We employ photolithography with backside alignment and deep silicon etching to prepare the mold. The mold is then filled with PDMS and cured. The silicon mold is then removed through a combination of wet and dry etching processes. The initial results, as exemplified in Figure 1, demonstrate the feasibility of the fabrication process. The process is currently being refined to achieve, more precise control of surface quality and to minimize the tendency of the fibers to adhere to each other during the release process. Detailed mechanical testing will be reported in the near future.

References

Figure 1: SEM image of PDMS fibers with inverted “L” shape.
High Sensitivity Uncooled Microcantilever Infrared Imaging Arrays

CNF Project # 1202-04

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Abstract

Multispectral Imaging, Inc., is developing and manufacturing infrared focal plane arrays (FPAs) of 160 x 120 pixels, based on an advanced bimorph microcantilever design. The bimorph design utilizes a combination of metallic and dielectric materials to create a temperature-sensitive structure that serves as the moving element in a variable-plate capacitor. The microcantilevers are integrated directly onto a complimentary metal oxide semiconductor (CMOS) integrated circuit, and all microcantilever materials are compatible with standard silicon IC foundry processing.

Summary

Uncooled vanadium oxide (VOx) and amorphous silicon (a-Si) microbolometers are presently the technologies of choice for thermally sensing and imaging long wave infrared radiation. However, the performances of these devices have not improved significantly in recent years and studies indicate that these technologies may be reaching their performance limits [1, 2].

Our proposed technique makes use of MEMS structures that respond mechanically to the absorption of infrared radiation. These structures were invented at the Oak Ridge National Laboratory (ORNL) [3] in the mid-1990s. Multispectral Imaging, Inc., (MII) has licensed the ORNL technology [4-6] and is pursuing its commercialization using the resources at Cornell NanoScale Science and Technology Facility (CNF).

Figure 1: Schematic diagram showing the operating principle of the bimorph microcantilever IR sensor.

Figure 2: Scanning electron micrograph of a portion of 160 x 120 MEMS imaging array. Inset: Close-up of a pixel in same array. Released height of the sensor structure is controlled using tensile and compressive stresses in the thin film layers used to fabricate the microcantilever.
Each pixel of our sensing array comprises the following components: an anchor, which elevates the sensing element above the substrate; a thermally isolating dielectric element of the paddle support arm; a thermally sensitive bimorph element of the paddle support arm; and the paddle itself. The gap between the paddle and the substrate serves as a resonant cavity for infrared radiation, and enhances the absorption of energy by the paddle. Heat flows from the paddle to the relatively cooler substrate through the thermally sensitive part of the support arm, causing the support arm to bend and the paddle to change its height relative to the substrate. The heat sensing bimorph microcantilever structures are fabricated directly onto the CMOS control and amplification electronics to produce a high performance, low cost imager.

MII has fabricated the first batches of fully integrated 160 x 120 FPAs at CNF with typical pixel functionalities ranging from 80% to > 95%. MII has an ongoing development program with Dalsa Semiconductor to commercialize this technology and we expect pixel yields of > 99% when in full production. Positional responsivities of greater than 0.3 μm/K have been modeled and measured for 50 μm pitch pixels, corresponding to a temperature coefficient of capacitance, ΔC/C, (equivalent to TCR for microbolometers) above 30%/K. This responsivity, along with noise-equivalent capacitances in the sub-attofarad range and nominal sensor capacitances of 15 fF, give modeled noise-equivalent temperature differential (NETD) of < 20 mK for these devices.

Preliminary infrared imagery has been obtained with a recently fabricated imaging array, assembled camera and control system with NEDTs in range of 350 mK using f/0.86 optics.

References

Ultra-High Density MEMS-Based Interconnect for Wafer-Level Ultra-Thin Die Stacking Technology

CNF Project # 1260-04
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Abstract

This work describes a novel smart three axis compliant (STAC) interconnect targeted to revolutionize chip-to-chip and chip-to-board high-density three dimensional (3D) integration for ultra-thin Si dies (≤ 75 µm) at the wafer level. The STAC interconnect is a 3D-compliant interconnect which allows stacked ultra-thin chips to move or flex freely during operation with negligible stress imposed on the die. The work shows that these interconnects can possibly accommodate mismatches of board or package coefficient of thermal expansion (CTE) from chip CTE. STAC interconnects are fabricated using MEMS technologies to support super-fine-pitch (≈ 20 µm pitch) interconnection. These interconnects are batch processed and die containing them can be stacked either at the wafer-level or at the die-level.

Summary

The constant demand for higher performance and greater functionality from an integrated circuit (IC) package has propelled the semiconductor packaging industry to seek more novel concepts to build and package IC chips. The IC industry has consistently pushed the limits of miniaturization with the advancement in lithographical techniques. Today the IC packaging industry not only has successfully packed more transistors in a square centimeter but also has successfully packaged chips with different functionality into a single module/package. The interconnection of multiple chips in a package has very quickly moved from the x and y-direction to the x, y and z-direction. The latter type of chip integration is more commonly known as three-dimensional (3D) packaging or chip stacking technology.

STAC interconnects are MEMS-based electrical interconnect that are microstructurally engineered to accommodate the relative displacements between ultra-thin through silicon vias (TSV)-based Si chips and the substrates to which they are bonded without transferring significant stress to the die itself. These interconnects are fabricated from metal films engineered to be released from the substrate when a sacrificial release layer under them is etched away. This is accomplished by using two distinct stresses—compressive and tensile—through the thickness of a single metal film. The alternate stresses in the film will generate a moment causing these interconnect to lift-up/curl. The radius of curvature, R of the interconnect is given by the following equation [1]:

\[ R = \frac{Y'h}{G} \] (1)

where \( Y' \) is the biaxial modulus, \( h \) is the interconnect film thickness and \( G \) is the interconnect film stress range.

STAC interconnects were formed using a titanium tungsten (TiW) film. The TiW film was sputter deposited in a cryopumped CVC 601 DC magnetron sputter system. The film was deposited by varying the argon base pressure. The recorded results show the large stress gradient can be achieved with the TiW film. Films can be deposited with stresses ranging from -1GPa to +1GPa. The STAC interconnect lift-up/curl height can be controlled by varying the stress range built into the TiW film during deposition. The greater the stress range, the higher the lift-up/curl. To improve the electrical and thermal properties of the interconnect, a thin layer of Cu is deposited over the TiW film.
Figures 1 and 2 show successfully fabricated STAC interconnects using the microfabrication steps described in the previous paragraph. The total beam length is 150 µm with 25 µm of its length fixed to the substrate (fully covering the Cu pad), and the beam width is 25 µm. Each interconnect is spaced 50 µm (width wise) by 170 µm (length wise) pitch, bringing the total number of STAC interconnects on a 1cm² chip to 11,200. The total number of I/O exceeds the long term requirement of the International Technology Roadmap for Semiconductors (ITRS) 2005. Since these interconnects were defined with photolithography and dry-etch chemistry, the exact beams shown in Figure 1 and Figure 2 can potentially be spaced at 30 µm (width wise) by 155 µm (length wise) pitch with a new mask, bringing the total number of compliant interconnects on a 1cm² chip to beyond 21,000.

References
MEMS Fabricated Vapor Cells

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Abstract

Vapor cells for atomic probing/pumping have been fabricated using a double-side polished silicon (Si) wafer. The cavity is formed by a potassium hydroxide (KOH) through-etch in the Si, with windows formed by an anodic bonded Pyrex® wafer and by a low pressure chemical vapor deposition low stress (LPCVD LS) nitride membrane. A glass capillary is sealed to the cavity, then used to pump down and refill the vapor cell.

Summary of Research

Vapor cells for atomic probing were fabricated in bulk Si. Two cavities are required: one to form the probe region, and another to act as the connection to the pump system and the gas. A channel was fabricated between the two cavities. In addition, LPCVD LS nitride was used as the membrane to cap the cavities. Finally a Pyrex wafer was anodic-bonded to finish the process.

The process is shown in Figure 1. First, a 40 µm channel is formed on the top side of a double-side-polished (DSP) Si wafer using a KOH etch with an oxide mask. Next, a micron of LPCVD LS nitride is deposited to act as a mask and, later, as a window membrane. The nitride is patterned with squares with sides of several mm’s, so that the front and back side patterns overlap with the channel. These patterns are KOH etched until it is stopped by the nitride on the other side of the DSP wafer. Finally, a Pyrex wafer is anodic-bonded to the top side of the DSP wafer, enclosing the channel and the cavities.

The cavity formed by the glass and nitride is used to enclose the probe gas (such as xenon or rubidium). The cavity on the backside is separated from the other cavity by the nitride, but is open on the back side. This nitride membrane is broken, then a glass capillary attached using Torr Seal. The glass capillary is attached to a pump system and a cylinder of the probe gas. The nitride membrane is strong enough to withstand pressures down to at least $10^{-5}$ Torr. A picture is shown in Figure 2 of the device, just before anodic bonding to a Pyrex wafer.

Figure 1, top: Process flow.

Figure 2, bottom: Picture of devices just before anodic bonding.
Prototype Cantilevers for AFM Lateral Force Measurement

CNF Project # 1273-04
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Abstract

One of the major difficulties in calibrating the lateral forces measured with optical lever atomic force microscopy is in determining the lateral optical lever sensitivity. Novel cantilevers have been designed and fabricated to simplify this measurement and pave the way for accurate calibrated cantilevers in which normal and lateral forces (e.g. from friction) can be easily measured.

Lateral (Friction) Force Microscopy

Atomic force microscopy (AFM) is widely used today to image surfaces and measure nanoscale forces. The most common form of AFM relies on a laser beam reflecting off the back of a cantilever to provide feedback on interaction of the cantilever tip with the surface of interest. The two most common deflections measured during surface interaction are normal and torsional, as depicted in Figure 1 (a) and (b), respectively. To accurately convert the observed AFM cantilever deflections to normal force and friction using contact AFM requires both accurate cantilever spring constant (stiffness) calibration for both normal and torsional deformation, as well as the determination of the normal and lateral sensitivity of the optical lever. Calibration of the normal spring constant and normal optical lever sensitivity is relatively straightforward using the reference cantilever technique and suitable SI traceable cantilever artifacts [1]. Calibration of the torsional spring constant and torsional optical lever sensitivity, needed for friction measurement, is more difficult.

Lateral Force Microscope Calibration Using a Modified Cantilever

One of the most straightforward methods of applying a known torsion to a cantilever involves fixing a lever to the cantilever and applying a force to the end of the lever arm as
in the technique of Feiler, et al. [2]. More recently, a refined version of this technique was demonstrated by Reitsma [3] in which a cross piece was glued to a cantilever forming a double lever arm (Figure 2). Application of a series of forces at different lever arm distances were performed to more precisely determine the torsional optical lever sensitivity (by a factor of 5 over Feiler) and demonstrate the principles of the new method.

The device was patterned on silicon-on-insulator wafers using optical lithography and etched using deep reactive ion etching (DRIE). Back side alignment was used to match the handle chip to the front side pattern. Final release of the structure was accomplished with buffered oxide etch (HF).

The microfabricated prototypes are currently being evaluated for their ability to measure the torsional optical lever sensitivity precisely. Torsional spring constants, measured using a calibrated instrumented indenter, will also be compared to Euler-Bernoulli theoretical models based on dimensional measurements and material properties for these experimental cantilevers.

References
Nanofountain Probes for the Delivery of Molecular Inks

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Abstract

Nanofountain probes (NFPs) are atomic force microscopy (AFM) probes designed to pattern planar substrates with molecular inks in the 50 nm - 1 µm range [1]. They extend the “dip-pen nanolithography” (DPN) [2] mode of patterning, by increasing the writing speed and eliminating the need for disruptive re-inking and subsequent probe realignment. This is accomplished by supplying fluid ink from on-chip reservoirs through microchannels to the probe tips. A volcano-shaped aperture keeps the fluid ink as close as possible to the probe-substrate contact point, without allowing a real flow over the substrate. This is essential to achieving sub-100 nm lines and avoids the formation of outer menisci (droplets) being dragged over the surface, as is the case with nanopipette and apertured probe writing devices.

Summary of Research

The nanofountain probe (NFP) allows parallel writing of molecular inks with a linear array of 12 probes, fed by two reservoirs [3]. It contains silicon nitride cantilevers with embedded microchannels, linking the on-chip reservoirs with volcano-shaped ink delivery tips (Figures 1, 2). The chip is designed to fit standard AFMs. Its fabrication relies on a combination of bulk and surface micromachining, with the release of the chips performed by deep reactive ion etching (DRIE).

A third generation NFP fabricated at the Cornell NanoScale Science and Technology Facility addressed the limitations experienced by the second generation chips. The use of silicon-on-insulator wafers provided better control of the residual stresses in the cantilevers to reduce bending upon release from the substrate, facilitating the patterning process. This also increased the laser signal detected by the AFM, thereby improving the image quality obtained when scanning. The new design features deeper microchannels to allow the delivery of larger molecules as well as stiffer cantilevers. The stiffness of the 520 µm long cantilevers is now 0.46 N/m; previously they were 0.312 N/m. In addition, more robust fabrication processes were used, increasing the device yield to about 75%.

As with the previous fountain probes, the connectivity and sealing of the NFP’s integrated fluidic system was tested with a DI water-diluted fluorescent dye (Texas Red, Dextran). The results showed a good penetration of the dye along the entire path of the channels, from the reservoir to the tips.
With the application of positive voltage to the reservoir area of the nanofountain probe, it was possible to deliver proteins. 15 µg/ml anti-BSA IgG from rabbit in 10 mM HEPES buffer was deposited on 16-mercaptophexadecanoic acid (MHA)-coated gold surfaces using the fountain probe. As is the case with nanoparticles, the size of patterned spots increased with increasing tip-substrate contact time.

A variety of cleaning methods were attempted to prolong the use of the nanofountain probes for protein deposition. Heating in air at 500°C for 10 minutes was able to partially remove some protein or salt residue at the probe tips. This technique was more useful for making slight adjustments to the curvature of the cantilevers. Immersing used NFPs in piranha solution (7:3 v/v H₂SO₄:H₂O₂) was the most effective method for removing residues from the tip area.

References


The new NFPs were used to make reproducible patterns of deoxyribonucleic acid (DNA) at room temperature in a relative humidity range of 20-90% (Figure 3). Thiol-modified oligonucleotides were patterned on a gold substrate and then hybridized to complementary nanoparticle-conjugated DNA strands, thereby confirming the biological activity of the patterned molecules. The successful patterning was verified by dark-field optical, AFM and SEM imaging.

In the interest of demonstrating patterning capabilities not easily achieved with other sub-100 nm patterning techniques, citrate-stabilized Au nanoparticles, with an average diameter of 15 nm, were directly patterned on SiO₂ substrates functionalized with 3-aminopropyltriethoxysilane (APTES) [4]. The negatively charged particles were electrostatically attracted to the positive NH₂ groups of APTES-SiO₂ substrates. The tapping mode AFM image in Figure 4 illustrates an array of dots patterned by the NFP. Topographic imaging of the patterns showed that the nanoparticle spot heights ranged from 10-30 nm depending on the density of the nanoparticles within the spots. This is in good agreement with the 15 nm average diameter of the nanoparticles. Typical patterned spots obtained with a tip-substrate contact time of 2s were 600-700 nm in diameter. As expected, spot sizes increased with increasing tip-substrate contact times. Scanning electron microscopy (SEM) imaging also confirmed the direct delivery of the nanoparticles. Computer simulations of fluid flow through the NFP indicated that the fluid remained confined to the core tip area of the probe, producing high-resolution patterns.
Air-Coupled Acoustic Method for Testing and Evaluating Micro-Scale Structures

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Abstract
Using an air-coupled transducer to externally excite and a Laser Doppler Vibrometer/Interferometer (LDV) to capture transient displacement waveforms, a technique to determine mechanical properties of micro-scale structural elements is created. The resonance frequencies and mechanical properties (Young’s modulus and stiffness) extracted from the transient displacement waveforms have been compared, with good agreement, to computational and simplified analytical models for commercially available micro-cantilever beams and micro-scale rotational oscillators fabricated for this study at CNF. The technique could serve to diagnose stiction problems of micro-scale structures.

Summary of Research
A non-contact, non-destructive testing and mechanical characterization method based on air-coupled acoustic excitation and transient displacement measurement using a Laser Doppler Interferometer/Vibrometer (LDV) for the determination of mechanical properties of micro-scale structures has been created (Figure 1). Using an air-coupled transducer in conjunction with a LDV in room conditions without contacting the micro-scale structure nor its substrate, a structural element is excited and induced vibrations are measured. Modal parameters (resonance frequencies) can be extracted from the transient displacement waveforms and compared with computational predictions such as a finite element (FE) analysis. As functions of material and geometric properties of the micro-scale structure, the modal properties allow the back-calculation of various mechanical properties, including stiffness and Young’s Modulus, from the measured transient displacement waveforms. The resonance frequencies of micro-scale structures can be excited with air-coupled transducers and transient responses can be acquired optically for the determination of mechanical properties such as Young’s modulus and stiffness.

Analytical and computational resonance frequencies of the micro-cantilever beam (Figure 2) and micro-scale rotational oscillator (Figure 3) oscillator are compared with experimentally determined frequencies with good agreement. An experimentally determined fundamental
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A resonance frequency of 187.5 kHz for the micro-cantilever beam was calculated from transient displacement data. This approximation was within the 20% dimensional tolerance allotted by the beam’s manufacturer dimensional specifications. An analytical calculation (from $E = 169 \text{ GPa}$, $\rho = 2,330 \text{ kg/m}^3$, and the reported geometry) showed 215.8 kHz (within 0.6% of the 217 kHz declared by the manufacturer and within 13.2% of the experimentally determined fundamental resonance frequency). The Young’s modulus of the micro-cantilever beam was found to be 127.5 GPa with a stiffness of 42.4 N/m (assuming the specified dimensions) was experimentally determined. The experimentally determined Young’s modulus (127.5 GPa) is significantly lower than the value provided by the manufacturer (169 GPa); assuming no dimensional variations, this deviation (24.5%) may be attributed to material property alterations such as oxidation of silicon and is unaccounted for in the FE model and/or analytical formulas.

For the micro-scale rotational oscillator, a fundamental in-plane resonance frequency of 240.5 kHz and 242.5 kHz was experimentally determined for different devices, within 10% of the 267.0 kHz and 266.4 kHz calculated by the FE analysis and a simplified analytical model (from $E = 175 \text{ GPa}$, $\rho = 2330 \text{ kg/m}^3$, and the device geometry), respectively. The resonance frequency of the first out-of-plane mode was identified at 888.0 kHz and 888.7 kHz (Figure 4) for two samples within 2.5% of the 878.8 kHz and 868.8 kHz calculated by the FE and simplified analytical models, respectively. A Young’s modulus of 182.3 GPa and an out-of-plane stiffness of 306.1 N/m were determined from the measured fundamental resonance frequency. The experimentally calculated Young’s modulus (182.3 GPa) for silicon is within 4.2% of the approximated Young’s modulus (175 GPa) and the experimentally determined out-of-plane stiffness is within 4.2% of the expected 293.9 N/m (based on $E = 175 \text{ GPa}$, $\rho = 2330 \text{ kg/m}^3$, and the device geometry).

Young’s modulus, and stiffness of micro-scale structures with known geometries can be effectively determined using air-coupled excitation in conjunction with a LDV. In addition, stiction problems for a particular micro-scale structure through the comparison of substrate and structure responses to the air-coupled acoustic field can be diagnosed. The method seems easily adaptable to various micro-scale structures. The proposed excitation and measurement mechanism features: simple set-up, function at room conditions, non-contact and non-destructive operations, and repeatable and rapid turnaround time for the evaluation of modal parameters and mechanical properties of microscale structures. The results could lead to a practical method for the evaluation of modal and mechanical properties of micro-scale structures using this non-contact and nondestructive technique.

Acknowledgements

The authors acknowledge Army Research Office (U. S. Army W911NF-05-1-0339) for their partial financial support for this project. The optical equipment used in the experiments were acquired through funding from the National Science Foundation (Nanoscale Exploratory Research (NER) Program, Award ID 0210242), and New York State Office of Science, Technology and Academic Research (NYSTAR). The fabrication of the micro-scale rotational oscillator was performed at the Cornell NanoScale Science and Technology Facility (CNF), a member of the National Nanotechnology Infrastructure Network of the National Science Foundation (Grant ECS 03-35765).

This report is extracted from our paper “Air-coupled acoustic method for testing and evaluation of micro-scale structures” from the journal Review of Scientific Instruments (Volume 78, ID Number 055105, May 2007); a full discussion of our method is available there.
Design and Fabrication of Silicon-Pyrex Micromixers for Enhanced Mixing

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Abstract
Silicon microelectromechanical systems (MEMS) technology was used to successfully fabricate a proposed multichannel micromixer and a standard T-junction micromixer for mixing enhancement study. Packaging via anodic bonding of the structured silicon substrates with Pyrex® was performed to facilitate optical and fluidic access to the channels of the micromixers. This packaging produces the desired triple-stacked (Pyrex/silicon/Pyrex) multi-channel micromixer and double-stacked (Pyrex/silicon) single-channel T-junction micromixer. Serving as a foundation for the fabrication of these mixing devices and the complementary experimental work was our prior numerical study carried out to evaluate, characterize, and optimize different model-based microchannel mixers. Our present work focuses on the testing and characterization of these fabricated devices for the ultimate purpose of designing efficient micro-channel mixers for microchemical systems applications.

Summary
Improvement of mixing quality in microchannel mixers/reactors, most especially for liquid/liquid multiphase reactions, has been recognized as a relevant technical issue critical to the development and application of integrated microchemical processing systems. The ineffective mixing in microchannel mixers/reactors, primarily due to the inherently diffusion-dominated laminar flow that characterizes such small-volume space, has become an issue of significant interest to many investigators working in the field of microreaction engineering.

The goal of our research study is to investigate mixing enhancement in microchannel mixers, through a theoretical as well as an experimental mixing study of currently utilized as well as proposed micromixing configurations. The design of our proposed micromixers for mixing enhancement is based on the mechanisms of fluid multilamination and elongational flow. In the mixing configurations designed, mixing is enhanced by placing static or passive mixing structures on the mixer channel floor to reduce the fluid diffusion path while at the same increasing significantly the fluid contact areas via creation of folding as well as local and global re-orientation of fluid interfaces.

The outcome of our prior numerical study using computational fluid dynamics (CFD) approach [1] shows that the proposed mixing configurations exhibit remarkably better mixing performance when compared with the standard T-junction micromixer. However, for our experimental mixing study, we select the standard T-junction micromixer (TJM) and one of the proposed mixing configurations that we refer to as multilaminated/elongational flow micromixer-4 (MEFM-4) based on the set criteria of minimum pressure drop and high mixing performance.
The completion of the fabrication aspect of our work, excluding packaging, has been reported earlier. Utilizing the silicon MEMS microfabrication technology [2], made available by the state-of-the-art equipment at CNF, the two above-mentioned mixing devices (designated as TjM and MEFM-4) were fabricated from silicon with Pyrex as the cover material. The fabrication process was achieved using silicon bulk micromachining techniques, which involved two basic steps; namely photolithography and deep reactive ion etching (DRIE). With these techniques, silicon wafers (double-side polished, p-type <100>, 4-inch diameter) were structured with fluidic channels of 300 µm deep. Silicon wafers of 500 µm and 800 µm in thickness were used for the fabrication of TjM and MEFM-4 respectively. Thicker wafers were used for fabricating MEFM-4 since deep structures of 300 µm each are required on both the front and back of the wafers.

It is worth mentioning that a three-step lithographic process is required to ensure structural and perfect back-to-front-alignment during the fabrication of the multi-channel MEFM-4 while a two-step lithographic process is needed for the single-channel TjM.

The DRIE recipe, enabled by inductively coupled plasma (ICP) was utilized for the deep etching of the mixing channels with vertical walls as well as the etching of the critical four through-holes in the MEFM-4. As shown in Figure 1, these 500 µm etched-through holes were designed to aid the transport of one fluid from the back-side inlet manifold to meet and mix with the second fluid coming from the front-side inlet manifold.

The packaging aspect of this work was recently concluded with the following processing steps: drilling of holes through Pyrex; anodic bonding of structured silicon with Pyrex; and dicing of the stacked wafers into individual micromixing chips. For fluidic access into MEFM-4, three holes (two inlet ports and one outlet port) were drilled into one of two Pyrex wafers to be bonded on both sides of the structured silicon wafer using a sonic mill. Anodic bonding was performed using 500 µm thick Pyrex glass wafers to obtain a Pyrex/silicon/Pyrex and Pyrex/silicon stacks for MEFM-4 and TjM respectively. The bonded wafers were then diced into individual micromixers of sizes 3.50 cm x 2.60 cm and 6.45 cm x 2.54 cm for MEFM-4 and TjM respectively (see Figures 2 and 3).

These micro-scale mixing devices, shown in Figures 4, are being tested and characterized for their relative mixing performance in our experimental set-up at New Jersey Center for MicroChemical Systems (NJCMCS) specifically designed for that purpose. In essence, our plan is to validate experimentally our numerical study on mixing enhancement using these fabricated devices.

References
Aqueous Transduction of Poly-SiGe Disk Resonators

CNF Project # 1380-05

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Abstract

This paper demonstrates an electrostatic transducer for lateral contour-mode resonators in which the transduction gaps are filled with a liquid dielectric (water) having much higher permittivity than air ($\varepsilon_{\text{water}} = 80.1$). Aqueous transduction is more efficient than air-gap transduction (lower motional impedance) and has a larger frequency tuning range than solid-dielectric transduction. We demonstrated a 42 megahertz (MHz) poly-SiGe disk resonator with deionized (DI) water confined to the electrode gaps. The resonator has a measured quality factor ($Q$) of 3,800, motional impedance of 3.9 k$\Omega$, and 3% series frequency tuning range.

Summary

Sounart and others have demonstrated that by using an local oscillator (LO) signal that is faster than the response time of a polar fluid, it is possible to prevent electrode polarization and double-layer formation, enabling electrostatic transduction in liquid media [1, 2]. To determine whether the same approach enhances the performance of contour-mode radio frequency micro-electromechanical systems (RF MEMS) resonators, we used a air-gap poly-SiGe disk resonator ($f_0 = 49.2$ MHz, $Q_{\text{air}} = 5,300$, $R_s = 510$ k$\Omega$ @ $V_P = 5V$) (Figure 1) [3] and submerged it in a water droplet. However, immersing the resonator in water caused excessive mass-loading and $Q$ losses resulting from viscous drag. To eliminate mass-loading and viscosity effects of the water droplet on the resonator, we coated the resonators with a hydrophobic self-assembled monolayer (SAM). The SAM is non-conformal, coating the top surface of the poly-SiGe disk resonator while leaving the 60 nm transducer gaps hydrophilic.

We placed a drop of DI water on the resonator and then slowly tipped the chip to one side to let the water droplet roll off the structure. The DI water ‘wicked’ the electrostatic transducer gaps (Figure 2a-b) and resonator performance improved to $Q$ of 430 at 36 MHz due to reduced viscous damping (Figure 2c). This low $Q$ resulted from water remaining underneath the
resonator after the droplet rolled off. Even though the gap under the disk was < 2 µm, water underneath would cause viscous damping and degrade the quality factor. To reduce this effect, we placed the water droplet a few microns away from the resonator and tipped the chip, letting the water droplet roll over the disk resonator (Figure 3a). The short time that the water droplet overlapped the resonator was enough to wick the transduction gaps but greatly reduced the chance of water seeping under the resonator. We were able to repeat the measurement multiple times with similar results. All measurements gave $Q > 3,500$ and $R_x < 4.2$ kΩ near 42 MHz (Figure 3b). The 100× improvement in motional impedance from air-gap-to-water is comparable to previous solid-dielectric transduced resonators [4, 5]. The experimental $R_x$ improvement is smaller than the theoretical enhancement (~ 3000×) due to the loss tangent of water, which adds to the total series impedance of the resonator.

References

Mechanical Coupling of 2D Resonator Arrays for MEMS Filter Applications

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Abstract

We present a study of mechanical coupling in two dimensional (2D) resonator arrays for filter applications. A robust coupling design for 2D array filters, comprised of weak coupling in one dimension and strong coupling in the second, is demonstrated experimentally and compared with weakly coupled and electrically summed 2D resonator array filters. Effects of inherent disorder in resonator arrays due to fabrication variations are minimized in this mechanical coupling scheme, averaging over resonator mismatch to form a smooth pass-band. The strongly-coupled 2D filter improves insertion loss and ripple without degradation in filter shape factor or stop-band rejection relative to its 1D counterpart.

Introduction

Though two-pole filters currently dominate radio frequency microelectromechanical systems (RF MEMS) filter research, there is an impetus to extend to multi-pole filters. As shown by Wang et al [1], increasing the number of resonators in a 1D filter improves both pass-band shape factor and stop-band rejection. However, spatial decay in the resonators and fabrication variations result in increased insertion loss and distortion in the passband as more resonators are added to the 1D array. This phenomenon has previously been investigated by Castanier and Pierre [2], using classical perturbation theory to model the effects of both dissipation and variations on 1D filters.

To improve this passband distortion, Judge et al. [3] proposed a 2D coupling which averages out the stochastic resonator characteristics. The design strongly couples an array of identical 1D filters, generating a two-dimensional matrix of resonators which are coupled weakly in one direction and strongly in the other (Figure 1).

Filter Design and Fabrication

In this study, four resonator coupling configurations are investigated to determine the effectiveness of the 2D strongly coupled array filter. We construct a 1D 4-pole filter (Figure 2a) as a basis of comparison for all 2D filters in the study. The performance of this 1D filter is compared with a set of four 1D 4-pole filters, electrically summed in parallel (Figure 2b), a 2D 4 × 4 array of resonators, weakly coupled in both directions (Figure 2c), and a 2D 4 × 4 array of resonators, coupled weakly in one direction and strongly in the other (Figure 2d).

The filters are composed of extensional wine glass ring resonators [4] with a fundamental resonance designed for 500 megahertz (MHz). The resonators are driven and sensed with lateral dielectric transduction [5]. The filters are fabricated in a simple silicon-on-insulator (SOI) process shown in Figure 3. First, 100 nm of low pressure chemical vapor deposition (LPCVD) stoichiometric silicon nitride are deposited on a 3 µm thick n+...
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Figure 4b presents the 50 Ω terminated S21 transmission of the four electrically summed 1D 4-pole filters (Figure 2b). The increased transduction area of the electrically summed 1D filters improved the insertion loss (IL, defined here at the maximum peak) by 7 dB relative to the single 1D chain. Additionally, the passband flattened to only 1.1 dB ripple, due to the summation of four 4-pole filters, offset in frequency from one another due to fabrication variations. However, these improvements are at the expense of filter shape factor and stop-band rejection. The electrically summed array filter has a 3 dB-8 dB shape factor of 2.19—a 30% degradation from the single 1D chain shape factor of 1.68. Furthermore, the stop-band rejection of the electrically summed filter reduces to 11.1 dB from 16.7 dB in the case of the single chain.

We next inspect the case of the 4 × 4 array of resonators, weakly coupled in both directions, shown in Figure 4c. The behavior of this multi-mode 2D array demonstrates that the strong coupling in the next filter is indeed strong enough relative to the weakly coupled direction.

Finally, we observe the effects of coupling a 4 × 4 2D array of resonators weakly in one direction (defining the resonant modes which contribute to the passband) and strongly in the other direction (averaging out fabrication variations). The unterminated frequency response of this filter is presented in Figure 4d. As in the case of the electrically summed filters, the insertion loss improves due to increased transduction area.

The ripple of the 2D strongly coupled filter improves from 5.4 dB to 4.2 dB relative to the 1D 4-pole filter. This corresponds to a 22% improvement in passband ripple. The improvement in passband distortion does not sacrifice filter stop-band rejection and shape factor. The stop-band rejection increases from 16.7 dB to 17.6 dB while the filter shape factor (3 dB–8 dB) decreases from 1.68 to 1.55 for the 2D strongly coupled filter relative to the 1D filter.

Conclusion

In this work, we demonstrated the effectiveness of a 2D mechanical coupling configuration for filters in reducing passband distortion due to micro-fabrication variations. A 2D filter comprised of a 4 × 4 array of bulk-mode wine glass ring resonators was demonstrated at 511 MHz. The 2D coupling provided a 22% improvement in unterminated passband ripple relative to its 1D counterpart, without degradation in stop-band rejection or shape factor. The 2D strong mechanical coupling configuration examined in this work can be implemented with any resonators in any fabrication process, providing more reliable and repeatable high-performance MEMS filters.

References

Directed Fluidic Assembly of Microscale Tiles

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Abstract

The aim of this project is to direct the assembly of micron-scale units (microtiles) into programmable, reconfigurable structures. Here we describe the fabrication of latching silicon microtiles for directed fluidic assembly. We also describe experiments in which fluid flow through a polydimethylsiloxane (PDMS) fluid chamber is controlled in order to direct the manipulation and assembly of various structures. In addition, we present simulation results which predict the further assembly capabilities of this system.

Summary

In order to manufacture increasingly integrated devices with incompatible component fabrication processes, reliable micro-/nano-assembly techniques are required. As size scales decrease, however, traditional pick-and-place assembly with motion planning becomes increasingly intractable. In response to these impediments, many “bottom-up” strategies have been developed, and have achieved the assembly of periodic, random, or specified target structures using pre-programmed components. In many cases however, structures to be assembled are either not known a priori, or would require too many distinct pre-programmed components to be assembled efficiently. Here we present an alternative approach to assemble arbitrarily-shaped microstructures from regular components on the micro scale. We circumvent the difficulties of pick-and-place microassembly by manipulating the components indirectly in a microfluidic chamber and relying on passive alignment and latching mechanisms to complete the assembly. We anticipate this new microassembly approach will form the basis for an alternative microfabrication paradigm and the manufacture of complex, integrated microsystems.

Experiments

Building on previous fluidic assembly experiments [1, 2], we achieved the automated manipulation and assembly of silicon microtiles. This was achieved by controlling the fluid flow conditions within and assembly chamber by regulating inlet and outlet conditions. As a first step, we introduced a single tile into the chamber and marched it along the substrate from one outlet position to the next. The next fundamental step towards the assembly of complex shapes was to create two- and three-tile assemblies (see Figure 1). In order to achieve this, we essentially rolled the tiles back and forth across the substrate together until the conditions were right for assembly. The resulting assembly rates are compared in Figure 2.
Simulations

In order to demonstrate directed assembly of microtiles, we carried out computational fluid dynamics simulations using the commercial software FLUENT. The results of these simulations, showing the assembly of a ‘C’ structure using square building blocks, are shown in Figure 3. Initially, the microtiles float neutrally buoyant in a chamber. The bottom of the chamber contains valves that can be selectively opened or closed and can be used to manipulate tile motion. When a valve is opened, there is fluid motion in that direction, which in turn causes a tile to move to that location. In this manner, tiles can be arbitrarily assembled at the bottom of the chamber, and the assembled tiles can be released back into the chamber by reversing flow at the substrate.

Fabrication

Solid 500 × 500 × 30 µm silicon tiles with patterned sides for self-alignment and passive latches for assembly (Figure 4) were designed using the L-Edit CAD software. Masks were created using the Heidelberg DWL 66 laser pattern generator. The tile patterns where then transferred to Shipley SPR 220-7.0 negative photoresist spun onto a silicon-on-insulator (SOI) wafer using the EV 620 contact aligner. After developing the resist, the exposed wafer was etched through the 30 µm device layer to expose the insulator using the Unaxis 770 Bosch etcher. A 49% HF solution bath was then used to release the etched tiles which were collected in a filter.

The directed assembly microfluidic chamber with on-chip valving was fabricated by multilayer PDMS soft lithography [3]. Soft lithography moulds were created by spinning SU-8 2050 photoresist on silicon wafers at 4000 rpm to obtain a ~ 40 µm thickness film which was patterned with contact alignment and hard baked for durability. Sylgard 184 silicone elastomer base and curing agent were used in a ratio of 20:1 for the thin fluidic layer and 5:1 for the thick pneumatic layer. The fluid layer was spun over its mould at 1250 rpm for a thickness of ~ 60 µm, while the pneumatic layer was poured approximately 1 cm thick. Both were cured at 80°C for one hour, then bonded together (after punching the pneumatic layer ports), and cured for several hours at 80°C. The fluid layer ports were then punched and the entire chamber was cleaned in an oxygen plasma and bonded to a glass slide.

Conclusion

We have presented a novel microfabrication approach which allows the assembly of non-regular microstructures from regular components on the microscale. Experiments have demonstrated the effectiveness of this technique in the assembly of two- and three- component structures. Simulations have further demonstrated the ability of this approach to assemble larger, non-regular structures. Since our assembly mechanism does not rely on unusual properties of the materials used, a wide range of component materials and assembly fluids is possible, along with a range of components sizes. All of this leads to a new microassembly approach for the manufacture of new, integrated microsystems.

References

Development of Nano Electromechanical Devices for Quantum Behavior Studies

CNF Project # 1480-06
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Abstract

Cooling mechanical resonators is currently a popular topic in many fields of physics including ultra high precision measurement and the study of transition between classical and quantum behavior of mechanical systems. The goal of this project is to fabricate nanomechanical devices coupled to a cavity Quantum ElectroDynamic (cQED) system. The photons from the cavity interact with and cool down the mechanical devices to their quantum ground state.

Summary

In this project we attempt to cool a mechanical device to its ground state using back action from a microwave cavity. We have designed and fabricated nanomechanical-resonator (NR) doubly clamped beams for magnetomotive measurement. The NR beams, fabricated on a 50 nm low stress low pressure chemical vapor deposition (LPCVD) silicon nitride layer, are written by electron beam lithography and are 50 nm wide and separated from a gate electrode by < 100 nm. The beams are released by two consecutive reactive ion etch (RIE) processes, one to vertically etch the nitride layer and the second to isotropically remove the silicon underneath the beams.

In the next phase of the project (work in progress), we will couple the NR beams to a microwave cavity formed from a 2.4 cm long coplanar waveguide. The resulting NR-cQED system will be installed on a 7 mK dilution refrigerator to cool the NR-beams to their quantum ground state. We will investigate ground-state squeezing of these mechanical beams to below the Heisenberg limit on continuous position detection.

References

Figure 1: SEM image of 150 nm wide beams.

Figure 2: SEM image of a suspended 50 nm wide beam.
Feasibility Study on the Microfabrication of MEMS Intergrated Antenna

CNF Project # 1482-06
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Abstract
An annular slot antenna has been constructed on microwave laminate. Two radio frequency microelectromechanical system (RF MEMS) switches are placed across the outer slot to connect/disconnect the ground and the inner strip. One switch is placed along the feeding microstrip line on the back side of the antenna and it allows the selectivity to feed either slot. When no switches are activated, the outer slot is fed to radiate at 2.4 gigahertz (GHz); when all the three switches are activated, the inner one is fed to radiate at 5.3 GHz. The frequency selection is realized by controlling the on/off states of the RF MEMS switches.

Summary of Research
Next-generation wireless communications in both commercial and military applications are increasingly moving to small, light-weight, and high-frequency systems with increased functionality and reduced power consumption. A concurrent trend in communication is to develop multi-mode/band systems on a single platform. These communication systems are becoming ever more complex with dozens of standards, a mixture of analog and digital formats and a diversity of spectral utilization. In many cases, these communication systems need to be supported by a number of antennas, which often radiate different frequency bands, polarizations and radiation patterns.

For a multi-mode/band system on a single platform, if one antenna is devoted to each mode/band operation, the limited space quickly becomes littered with antennas, and the use of dedicated antennas is costly, leads to unwanted proximity-coupling and degrades the overall performance. One solution, which keeps the entire system compact and satisfies multi-mode/band operations, is to design a single antenna that can be reconfigured or tuned to deliver the desired frequency bands, polarizations and radiation patterns without sacrificing radiation efficiency.

The radiation behavior of an antenna depends on the distribution (path and electrical length) of the resonant currents over the antenna surface, which is further determined by antenna geometrical parameters, such as size, shape and position of radiating and parasitic elements. Reconfiguring these parameters enables multiple functions to be performed with the same antenna structure. A number of approaches have been proposed for implementing reconfigurable antennas [1-4]. Most of these approaches make use of switches or varactors to change the physical dimensions of an antenna structure or the distribution of the antenna resonant currents.

Reconfiguring antennas can be realized by using semiconductor devices (PINs and FETs) and RF MEMS switches. For semiconductor devices, when the signal frequency becomes greater than 1 GHz, they have large insertion losses and poor isolation. They produce
significant intermodulation distortion as a consequence of their non-linear I-V feature. The antennas made by using these devices will be characteristic of high power consumption. RF MEMS switches are devices that use mechanical movement to achieve an on/off operation. They have very high isolation, very low insertion loss and very high linearity. The RF MEMS switches based on electrostatic actuation have near-zero power consumption. Being essentially broadband devices, RF MEMS switches are far less affected by frequency and are more suitable for high-frequency applications. Most importantly, the MEMS technology enables monolithic integration with antenna radiating elements. RF MEMS switches are now increasingly used to realize reconfigurable or tunable RF devices, components and systems. As RF MEMS switches are becoming mature, it is important to develop novel ideas and practical approaches to make full use of MEMS switches’ remarkable characteristics and to achieve novel, high-performance RF devices, components and systems.

In the previous year, we worked on a reconfigurable annular slot antenna. The antenna is designed to primarily realize frequency band selectivity while maintaining radiation pattern. Slot antennas are attractive for many communication and radar applications due to geometric simplicity, efficiency, reliability, and light-weight. It is known that an antenna alters its radiation characteristics when its physical geometry is changed. The change here is realized by RF MEMS switches that are located at the annular slots and along the signal-feeding microstrip line. Figure 1 shows the front side of the annular slot antenna constructed on a TMM10i ($\varepsilon_r = 9.8, \tan\delta = 0.002$) Rogers microwave laminate. Two switches are placed across the outer slot to connect/disconnect the main ground and the inner metal stripe. Figure 2 shows the double-arm DC contact RF MEMS switch used in this implementation. The dual band behavior is achieved by selectively activating the two concentric radiating slots. One RF MEMS switch is placed along the feeding microstrip line on the back side of the antenna and it allows the selectivity to feed either slot.

When no switches are activated, the outer slot is fed to radiate at a low frequency of 2.4 GHz; when all the three switches are activated, the inner one is fed to radiate at a high frequency of 5.3 GHz. Figures 3 shows the simulated and measured return losses corresponding to the two operation modes, respectively. As shown, the frequency selection is realized by controlling the on/off states of the RF MEMS switches. The measured frequency locations are in good agreement with the simulated results with a very small discrepancy at 5.3 GHz.

References


Fabrication of Ultra-Sharp Diamond AFM Probes

CNF Project # 1485-06
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Abstract

Monolithic, ultra-sharp ($R < 10 \text{ nm}$) diamond probes were fabricated from ultra-nanocrystalline diamond (UNCD®). The fabrication sequence developed uses optimized diamond processing with precision lithography, selective etching, anisotropic etching, and precision dicing. The probes showed low wear and stiction as expected, based on the physical properties of diamond, outperforming standard silicon nitride probes in these aspects. These all-diamond probes should open up new avenues of research to explore nanoscale friction and adhesion at the nanoscale, as well as nanofabrication.

Summary of Research

Ultra-sharp atomic force microscopy (AFM) probes with diamond tips are long sought after, but so far, not yet commercially available devices. The roughness of the diamond films and several integration difficulties accounted for this. Our ultrananocrystalline diamond (UNCD®) has been proven [1] to have the properties that make diamond AFM probes desirable: high hardness, low wear, and low stiction to particles, while having as low as 7 nm roughness ($R_a$), low differential stress, and good conformity. This material and its growth process are well-suited for fabricating tips. All-diamond probes are superior to coated silicon tips in terms of sharpness, since in the latter, the tip radius is given by the thickness of the diamond film. A preliminary optimization study showed that the probe fabrication process can be used to obtain tip radii as sharp as 5 nm (Figure 1), on the same order as the best silicon nitride probes on the market. Unfortunately, the as-fabricated tips face the silicon wafer, which makes it necessary to reverse them by attaching/transferring them to another substrate. While this process is standard for silicon nitride tip fabrication [2], difficulties arise when using diamond. The probe fabrication sequence we engineered was optimized for convenient etching of diamond, efficient transfer of the diamond layer and probes by bonding to a Pyrex® wafer, and final release of the structures. Ultra-sharp diamond AFM probes fabricated using this process can be seen in Figure 2.

Probes were produced both for tapping mode imaging (higher resonance frequency and stiffness) and for contact mode techniques (low stiffness, low resonance frequency). The characteristics of the probes are given in Table 1. The layout provided each carrier chip with four probes, two on each side of the chip, for maximization of the number of useful probes for testing.
Mechanical Devices

The probes were tested in standard AFM equipment (Veeco, DI MultiMode). Wear tests were performed [3] in conjunction with high resolution scanning electron microscopy (SEM) and tunneling electron microscopy (TEM) investigation of the tips, showing a net superior behavior of the diamond tips as compared with standard silicon nitride tips. The tests consisted in scanning/imaging for 100 times a 1 mm × 1 mm area of a UNCD surface with the diamond probes in contact mode with zero net force. While standard silicon nitride probes increased their contact radius from 12 to 75 nm in the given conditions, UNCD probes showed no radius increase outside of typical data scatter (± 5 nm).

Batch processing of ultra-sharp UNCD probes allows for developing new or enhanced scanning probe techniques by integration in complex probe MEMS structures, such as arrays of probes (for parallel imaging [4], data storage [5], parallel dip-pen lithography [6], etc.) or high cost specialized probes such as nanofountain-probes [7] and probes with integrated actuation and sensing [8], making such probes affordable by compensating costs with enhanced life time. Conducting UNCD can also be used to develop probes that use electrochemical contrast that have unparalleled dimensional stability as well as for scanning spreading resistance microscopy [1].

Probes will be sent out to users for beta testing. Interested users are encouraged to contact the authors for receiving evaluation samples.

Work was supported by the National Science Foundation through its SBIR/STTR program and the State of Illinois’ Department of Commerce and Economic Opportunity, and acknowledges fabrication of prototypes in part at the Cornell NanoScale Facility, a member of the National Nanotechnology Infrastructure Network, which is supported by the National Science Foundation (Grant ECS 03-35765).

References


Table 1: Characteristics of the produced probes.

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Microfabrication of Flexible Sensing Arrays: Active Nanoparticle Thin Films and Interdigitated Microelectrodes on Plastic

CNF Project # 1507-06
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Abstract

Traditionally most of the electronics fabrication work at CNF is based on rigid silicon and glass substrates. The goal of the Center of Advanced Microelectronic Manufacturing (CAMM) is to develop tooling and processes to use unsupported flexible polymeric substrates. Recently, C. J. Zhong [1] and his group have fabricated gold microelectrode devices on glass substrates to explore the use of thin film coatings of monolayer-capped nanoparticles for chemical sensors (volatile organic compound sensing), medical diagnostics and other microelectronic applications. Here we attempt to fabricate copper microelectrode devices using unsupported poly(ethyleneterephthalate) (PET) substrates for use in low-cost, disposable sensor applications.

Summary of Research

Most of the fabrication processes such as photolithography, etching, and stripping for building the copper interdigitated microelectrodes on PET substrates were performed at CNF. Pieces of 125 µm (5 mil) thick heat stabilized PET polyester (DuPont-Tejin Films Melinex ST507) film was isopropyl alcohol (IPA) and oxygen plasma cleaned followed by sputtering of 50 Å chrome and 3000 Å copper at Endicott Interconnect Technologies (EI). Four inch diameter “mock wafers” were cut from the Cu sputtered pieces of PET. Shipley 1813 photoresist was spin-coated at CNF to a thickness of 1.5 µm. The photolithography mask was designed and fabricated at CNF.

The CNF HTG System III-HR contact aligner with 365 nm wavelength UV light was used to image the microelectrode pattern on the resist coated PET substrate. Four seconds was determined to be the optimal exposure time. The resist was developed using AZ 300 MIF for one minute at room temperature. The line/space (in microns) feature size of the microelectrodes include: 5/5, 10/10, 10/15, and 15/15 µm. Figures 1 and 2 show 15/15 µm and 5/5 µm line/space features in the resist after developing. The CE-200 etchant used at CNF significantly over etched the features. Cupric chloride (concentration [175g/L]) was also used to etch the copper layer. The cupric etching process was done at EI, and over etching was reduced.

After the etching, the AZ-300T at CNF was used to strip the resist at room temperature. Figures 3 and 4 show the resulting flexible

Figure 1: 15/15 µm line/space resist features after development.

Figure 2: 5/5 µm line/space resist features after development.
copper microelectrodes on the PET substrate. Figure 3 shows the four inch flexible polyester “mock-wafer” with 1.5 mm Cu circuit lines. These large circuit features connect to the 15 µm sized (over etched) circuit lines shown at high magnification in Figure 4.

This research demonstrates the feasibility of fabricating the flexible copper microelectrodes on flexible unsupported PET substrates using the microfabrication facilities at CNF. Parameters such as etchant concentration, etching time and temperature need to be further improved to prevent overetching the copper layer. PET substrate surface flatness will be improved by use of tooling and a more carefully controlled Cu sputtering process.

The CNF provides education and training for the CAMM students as well as facilities to design and fabricate masks, test various materials, processes and tooling as the CAMM is being established. The long term objective of this project is to develop roll-to-roll microelectronics manufacturing technologies to fabricate large-area flexible chemical sensing arrays at the CAMM. The CAMM was established in 2005 when the United States Display Consortium selected Binghamton University, a global leader in electronics packaging and small scale systems integration, to spearhead development of next generation roll-to-roll electronics manufacturing capabilities [2]. A unique collaborative effort, the CAMM brings together Endicott Interconnect Technologies, Inc., Cornell University and other partners from government, industry and academia to tackle the myriad challenges of this emergent electronics manufacturing technology.

References


Microfluidic Devices for Insect MEMS

CNF Project # 1516-06
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Abstract
This work describes the development of an electroactive microwell based chemical/drug delivery system and its pupae stage implantation within Manduca Sexta moths. This forms one of the critical components in our development of Insect MEMS which aim to fuse nanodevice technology with living systems. The goal of this system is to provide “on-command” chemically induced immobilization and subsequent reanimation of the insect. In this talk we will present the results of our adult survivability data for late and early pupae stage microdevice insertion experiments, our initial toxin injection experiments, and a comprehensive numerical/experimental study of our electroactive drug delivery system.

Introduction
Modern microsystems technology has enabled the development of a large number of biomedical devices which allow us to monitor biological systems and biomolecular events with extreme precision. While such technology is proving extremely successful, rarely has the extension been made to exerting active control over a living system. In this work we present our initial results on the development of implanted microfluidic devices which enable such control over flying insects, namely Manduca Sexta Moths. One potential application of such a hybrid-insect system is in the development of microscale air vehicles, which exploit the highly evolved aerodynamics of insects with recent advances in microdevice engineering. An overview illustrating the system integration is presented in Figure 1.

Experimental Results
As shown in Figure 2, the microdevice platform is partially implanted within the insect at the dorsal thorax of the pupae. To find the optimal location (which minimizes its physiological footprint, maximizes survivability, and favorably disperses the toxin), both the platform size and implantation location were varied. Figure 1 shows the results of a successful emergence of an insect with fully developed wings.

Figure 3 illustrates an abbreviated version of the steps involved in our injection experiments used to determine the dosage effects on the degree and length of insect paralysis. For example, injection of 5 µL of a 5.839M...
solution of L-Glutamic acid potassium salt monohydrate, which comprises one of the major components of spider venom, into the thorax, the insect was immobilized for approximately 5 hrs, after which the insect regained its pre-injection activity level. The electroactive microwell drug delivery system developed here is based on the fusion of previous implantable drug delivery technologies with our recently developed electrokinetically active microwells.

Briefly, the drug is stored within a well, etched into a silicon substrate (similar to illustrated in Figure 1b) which is sealed from the exterior using a thin gold membrane on PDMS (polydimethylsiloxane). When a dosage command is issued, a voltage is applied to electrochemically dissolve the gold membrane, exposing the toxin. Unlike previous approaches, which rely on diffusive transport of the drug, we incorporate an electrokinetic injector whereby a potential field is applied between the top and bottom of the well and the drug is electroosmotically rejected from the well. A detailed, finite element based, model of the local fluid and transport dynamics involved in the drug ejection procedure will be presented along with our initial experimental results (see Figure 4). As will be demonstrated, this approach reduces the drug injection time from approximately 30 minutes for diffusive transport to less than 1s.
Capacitive Drive and Detection of MEMS
Resonant Motion: Electrical Integration of MEMS

CNF Project # 1520-07
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Abstract
Incorporation of microelectromechanical system (MEMS) oscillators into devices has been limited by the lack of an easily-integrated motional detection scheme. A capacitive detection scheme enabled by impedance matching techniques has been demonstrated for nanowires. However, no measurement had been demonstrated in micron-scale oscillators of interest in sensor and electrical filter applications. Through our work at the CNF, we have demonstrated entirely electrical drive and detection of micron-scale dome oscillators.

Summary
An easily-integrated readout mechanism would enable MEMS/NEMS oscillators to be incorporated to electrical devices, whose applications could include electrical filtering, ultra-sensitive inertial mass sensing [1], and any mechanical operations requiring electrical feedback. A purely electrical readout scheme, in place of the effective-but-bulky interferometric or magnetomotive techniques commonly employed, naturally lends itself to device integration. We are developing a technique that detects the fluctuations in capacitance between the oscillating MEMS/NEMS structure and a charged gate in its vicinity, as demonstrated for nanowires [2]. The tiny resulting signals can be effectively isolated and amplified through the use of an impedance matching circuit to match the oscillator to conventional 50Ω-electronics (Figure 1). Ultimately this approach could be integrated into complementary metal oxide semiconductor (CMOS) processes and devices, allowing far wider application to MEMS/NEMS technology.

The electrical readout technique is demonstrated using concave polysilicon domes. The single crystal silicon substrate and upper polysilicon device layer are insulated by a layer of thermal oxide. The oxide is removed by buffered oxide etch through photolithographically defined etch holes opened in the polysilicon in a reactive ion etch, resulting in a radially symmetric drum, dome, or bowl, depending on film stresses, whose upper and lower surfaces represent the plates of a capacitor (Figure 2). The dome oscillator and a neighboring gold bond pad are surrounded by a

Figure 1: Impedance matching circuitry diagramed with a cross section of a dome oscillator. The gate capacitance originates from the area of the bond pad and oscillator.

Figure 2: SEM of single device. A trench in the polysilicon electrically isolates the enclosed area from the rest of the wafer.
Mechanical Devices

trench through the polysilicon into the underlying oxide, electrically insulating the structure from the rest of the wafer and limiting the gate capacitance. Resulting devices are wire bonded to a ceramic DIP and connected to a printed circuit board containing the impedance matching circuitry. The ensemble is placed in vacuum for measurement.

Readout of MEMS/NEMS oscillators examines the vibratory modes of the a structure. We are able to identify identical modes with both laser interferometric and an entirely electrical readout (Figure 3 & 4). The electrical readout analyzes a reflected RF signal from the device driven by a network analyzer. At resonance, the fluctuations of the oscillator create an effective impedance, upsetting the impedance match. The electrical signal can be used to drive of the device as well as detect the motion of the oscillator.

References


Figure 3, top: Mechanical resonance peak of dome oscillator, driven and detected optically.

Figure 4, bottom: The same resonance peak, driven and detected with fully electric readout technique.
Arrays of MEMS Intertial Sensors with Optical Readout

CNF Project # 1538-07
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Abstract

Microelectromechanical system (MEMS)-based inertial sensors have many advantages over traditional discrete-component-based inertial sensor designs. This is due to their smaller size, batch fabrication, lower cost and higher robustness. Although MEMS-based inertial sensors can withstand high-g accelerations, they are prone to bias instability. This limits their current use to less demanding applications [1]. The goal of our project was to explore alternative technological approaches to MEMS-based gyroscopes, with potential of achieving the navigation grade performance.

Summary of Research

We have focused on devices that consist of double-tine resonators arranged into a tuning fork shape. Each device features two driving tines and two sensing tines (Figure 1). The driving (lower) tines are to be actuated optically with an amplitude of up to several micrometers. The Coriolis force is exerted on the upper tines when the system is being rotated around the main axis. This force can be detected by observing oscillation of the sensing tines, hence detecting the rotation of the system. The deflection is probed using the optical lever readout similar to the one used in atomic force microscopes [2] and which can detect sub-angstrom displacements.

We have fabricated several batches of devices with different geometries, all featuring single-crystal silicon (Si) as a structural material for the driving tines. Single-crystal Si was chosen as it is characterized by low mechanical losses. Sensing tines were fabricated using either Si (a device layer in SOI wafers) or low stress LPCVD silicon nitride (SiNₓ)

as a structural material. In order to fabricate our targeted devices, we have implemented a three-mask process flow that relies entirely on dry etching. The driving tines and the Si sensing tines were patterned using the deep reactive ion etching (DRIE, Bosch process), while SiNₓ sensing tines were patterned using the regular fluorine-based RIE.

Our preliminary characterization of the fabricated devices indicated resonant frequencies of driving and sensing resonators in the ranges of 8-25 kHz and 2-4 kHz, respectively. The resonators in vacuum exhibited typical Q-factors above 10⁴.

References

Figure 1: Photograph of one of the structures. Driving and sensing tines oscillate in perpendicular planes.

Figure 2: Array of inertial sensors.
Fabrication of Nanomechanical Resonators Using a Sequential Exposure of Opposite Tone Electron Beam Resists

CNF Project # STAFF
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Abstract

We report a method for fabricating nanomechanical structures from a hydrogen silsesquioxane (HSQ)-based negative tone electron beam lithography (EBL) resist. This technique utilizes a sequential exposure of poly(methyl methacrylate) (PMMA) and HSQ to produce self supporting nanomechanical resonators. The first exposure is performed in the PMMA layer. This pattern is developed, HSQ is applied to the substrate over the PMMA, and a second exposure is performed registered to the first. The patterned PMMA film serves as a structural template for patterns written in the HSQ layer. HSQ exposed in patterned regions of the PMMA film becomes mechanically connected to the substrate. HSQ patterns connected to the substrate at multiple points can be released by removing the PMMA film using an oxygen plasma. Nanomechanical oscillators of different lengths were produced using this technique and the resonant frequency spectrum of these structures was examined using an optical excitation and detection technique. Resonant frequencies from 6 to 24 megahertz (MHz) with quality ($Q$) factors between 300 and 1500 were measured and found to be a strong function of oscillator length. The dynamic response of the system was modeled using the Euler-Bernoulli formalism and found to agree with the experimentally measured results.

Introduction

In the past decade, research on nanomechanical structures [1,2] has illuminated fundamental physical phenomena, enabled the fabrication of mass sensors with unprecedented sensitivity, and yielded a host of other unique structures. In 2001, Tanenbaum, et al., presented a simple process for forming nanomechanical resonators composed entirely of a hydrogen silsesquioxane (HSQ)-based negative tone electron beam lithography (EBL) resist [3]. These structures were created by performing a double exposure of the HSQ layer using two patterns exposed at different beam energies: 30 keV and 1 keV. The 1 keV electrons do not penetrate the entire HSQ film; they are confined to the near surface region. In contrast, 30 keV electrons completely penetrate the HSQ film and enter the substrate. Tanenbaum capitalized on this difference in beam penetration depths and used the 1 keV beam to expose patterns of nanoscale resonators. Portions of the pattern were selectively re-exposed using a 30 keV beam. During the HSQ develop process, the regions patterned at 30 keV remained rigidly fixed to the substrate while the patterns exposed at the lower energy were undercut by the developer releasing them. Like Tanenbaum’s work, this process uses a double exposure. However, the first exposure is performed in a layer of poly(methyl methacrylate) (PMMA). Once this pattern is developed, HSQ is applied to the substrate over...
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the PMMA and a second exposure is performed registered to the first. The PMMA film acts as a structural template for patterns written in the HSQ layer. HSQ that is exposed in patterned regions of the PMMA film becomes “anchored” to the substrate forming a robust mechanical connection. HSQ patterns connecting multiple “anchor” points can be released by removing the PMMA creating self supporting structures.

Summary

An overview of the fabrication process is shown in Figure 1. 100 mm diameter Si (100) n-type wafers were used as the substrates for this work. An array of alignment mark structures was patterned onto these wafers using standard photolithography and dry etching techniques. These patterns consisted of an array of 10 µm wide squares etched 500 nm into the Si substrate. 500 nm of PMMA was spin-coated onto the wafers and baked at 170°C for 15 min (Figure 1-a). The substrates were exposed using a Leica VB6HR operated at 100 keV with a 5 nA beam current. The locations of the anchor points were exposed in the PMMA film using a dose of 1000 µC/cm². These patterns were developed in a solution of methyl isobutyl ketone (MIBK) and isopropyl alcohol, 1:3 (Figure 1-b). After inspecting the developed patterns, the substrates were coated in 4% HSQ in MIBK (Dow Corning, XR1541). This film has a nominal thickness of 120 nm when coated at 3000 rpm. The HSQ was applied to the PMMA using a static dispense process and allowed to puddle for 3 seconds before spinning. This puddle process facilitated coverage of the PMMA topography. Early experiments revealed that features in the PMMA layer less than or equal to 200 nm were almost completely planarized using this approach (Figure 1-c). After aligning to the predefined registration mark patterns, the locations of the anchor points were re-exposed. Patterns for structures that would ultimately become released or undercut were exposed such that they were connected to the anchor points. A dose of 2200 µC/cm² was used for these exposures. The HSQ film was developed using a commercially available 0.26 M aqueous solution of TMAH (AZ, 300 MIF). A 4 min immersion develop with no agitation followed by a deionized water rinse was used for this process (Figure 1-d). Following inspection of the developed patterns, the PMMA film was removed from the substrates using isotropic O₂ plasma etching (Figure 1-e). An oblique angle scanning electron microscope (SEM) image of a fully released bridge is shown in Figure 2-a.

Optionally, completed structures could be coated with a layer of Al using electron beam evaporation (Figure 1-f). Beyond changing the optical properties of the released structures, this coating provides a way to produce electrical interconnects and probing pads that are connected to the released nanomechanical structures and isolated from the substrate. An example of a completed Al-coated HSQ nanomechanical structure fabricated using this process is shown in Figure 2-b. Figure 3 shows nanomechanical bug-like structures that illustrate the compressive stress state residing within the HSQ layer.

References