

# Atomic Resolution Disk Resonant Force and Displacement Sensors for Measurements in Liquid

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**Abstract**—This letter presents resonant microelectromechanical systems capable of resolving subatomic features and/or measurement of atomic level forces in liquid media (in addition to air/vacuum). It has previously been shown that forces in the micro- and nano-Newton range applied to flexible extensional-mode resonant microstructures can cause significant resonant frequency shifts [1]. In this letter, the same principle has been applied to rotational mode disk resonators that are capable of maintaining relatively high quality factors in liquid. Preliminary results indicate about tenfold improvement in combined displacement-force resolution figure-of-merit compared with typical piezoresistive cantilevers when operating in liquid. Using integrated comb-drive electrostatic actuators, displacement and force resolutions as high as 200 fm and 1 nN, respectively, have been demonstrated. By application of the force through a levering mechanism, force sensitivities in the pN range can be achieved while maintaining sub-nm spatial resolution.

**Index Terms**—MEMS resonant force-displacement sensor, in-liquid measurements, atomic force microscopy.

## I. INTRODUCTION

**S**UB-ATOMIC displacement and force measurements are integral to modern biological sciences for the study of cell mechanobiology, interaction forces within various biomolecules, nanomechanical properties of biological samples, monitoring of protein dynamics and imaging biological samples in their native environments [2], [3]. To date, the most powerful tool to study biological systems is frequency-modulation atomic force microscopy (FM-AFM) utilizing optical lever and optical interferometry. Such techniques, however, require external sensing elements, making the overall system somewhat bulky, costly and complex. Moreover, due to significant quality factor drop of conventional AFM cantilevers in air and liquid (limited to 100 and 10, respectively), true atomic resolution imaging and force measurements in air and especially in liquid remain a formidable challenge [4]. Employing sophisticated instrumentation and readout electronics along with application of proper modifications within commercially available AFMs, subatomic displacement and force resolutions

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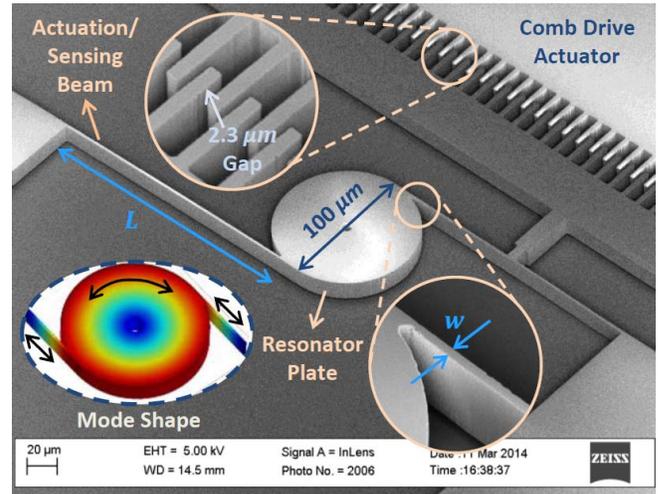


Fig. 1. SEM view of a 2.3 MHz in-plane disk resonant force-displacement sensor capable of operating in liquid. The inset on the bottom left depicts the finite element modal analysis of the rotational resonant mode of the disk structure prior to application of any external force.

have been demonstrated in liquid [4], [5]. For instance, using small oscillation amplitude (0.16–0.33 nm) of a  $\sim 40$  N/m cantilever, vertical resolution of 2–6 pm has been shown [5]. Piezoelectric and piezoresistive sensing, on the other hand, enable integration of sensing elements on the AFM cantilevers which significantly simplifies the experimental setup by eliminating the need for external sensing elements, precise system alignment and complex circuitry for operation [6]. However, such methods require complex fabrication processes and cannot compete with some of the conventional technologies in terms of sensitivity [6]–[8]. Taking advantage of high performance of microscale rotational mode disk resonators in liquid, this work presents an integrated and convenient (with fully electronic readout) force-displacement measurement technique that can be deployed in various environments (vacuum, air, and liquid) with higher sensitivities. Such resonant MEMS devices can be potentially transformed to AFM technology overcoming low  $Q$  limitation of currently available cantilevers in liquid.

## II. DEVICE FABRICATION AND DESCRIPTION

Figure 1 illustrates SEM view of one of the test structures used in this work that is comprised of a disk resonator coupled to a comb-drive electrostatic actuators. The structures are fully made of single crystalline silicon and are fabricated using a

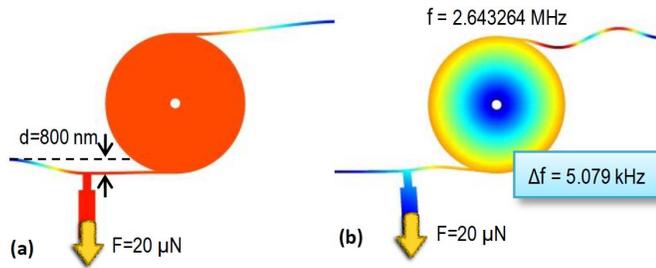


Fig. 2. Finite element analysis using COMSOL Multiphysics (a) static analysis of the 2.3 MHz disk resonant structure of Fig. 1 subjected to an external local force to one of its support beams. (b) Modal analysis of the same structure undergoing the same force showing  $\sim 5$  kHz negative frequency shift compared to no external force case. Red and blue show the maximum and minimum vibration amplitudes, respectively.

single-mask process on silicon-on-insulator (SOI) substrates with device and buried oxide layer thicknesses of  $15 \mu\text{m}$  and  $5 \mu\text{m}$ , respectively. The fabrication process includes silicon DRIE to carve the structures out of the silicon device layer, and releasing them by etching the underlying buried oxide (BOX) layer in hydrofluoric acid (HF). The long narrow tangential beams on the two sides of the disk, other than being structural supports, are responsible for both thermal actuation and piezoresistive detection of the resonator vibrations [9]. Upon periodic electro-thermal excitation of the resonator with the appropriate frequency, the disk starts vibrating in its rotational resonance mode (as depicted in the inset of Fig. 1). In other words, once the support beams vibrate in an extensional mode, the resonator disk plate periodically rotates back and forth. Since, in such mode, all the surfaces of the resonator slide in parallel to the surrounding environment, the interfacial interactions and in turn viscous damping, which is the dominant factor in liquid, are minimized. As a result, such devices can reach  $Q$ s as high as 300 when operating in liquid [9] which is a substantial improvement compared to e.g. flexural mode cantilevers with  $Q$ s below 10. The embedded comb drive actuator is used to simulate the effect of an external force applied to the resonant force sensor and has 55 interdigitated electrode pairs separated by photolithography defined  $2.3 \mu\text{m}$  wide air-gaps. The force from the actuator, conveyed through a linkage beam, can slightly deform the resonator structure (Fig. 2(a)), and consequently change its effective mechanical stiffness and therefore resonance frequency (Fig. 2(b)). The middle point of the support beam is chosen in this work as the contact point to cause considerable deformations within the structure without introducing excessive support loss to the structure.

### III. MEASUREMENT RESULTS AND DISCUSSION

To perform the tests in liquid, a square-shaped glassware reservoir ( $1 \text{ cm} \times 1 \text{ cm} \times 5 \text{ mm}$ ) was fabricated and fixed to the designated location on the PCB, where the device is placed in the middle of, and filled out with liquid after wire-bonding the resonator to the appropriate metal tracks on the PCB.

Resonant structures with different dimensions were tested (as described in [1] and [9]) in both air and xylene at room temperature ( $25^\circ\text{C}$ ) showing  $Q$  factors as high as  $\sim 4000$  and

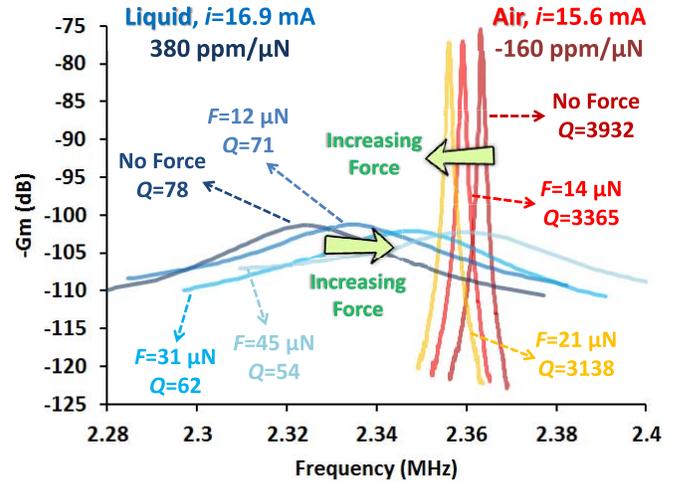


Fig. 3. Measured frequency responses for the 2.3 MHz thermal-piezoresistive resonator of Fig. 1 in air and xylene with different applied external forces. Red and blue plots refer to air and liquid testing conditions, respectively. The results indicate  $\sim 2\times$  higher force-displacement sensitivities in liquid than in air. Dielectric constant of  $\epsilon_r = 2.2$  for xylene has been used in electrostatic force calculations.

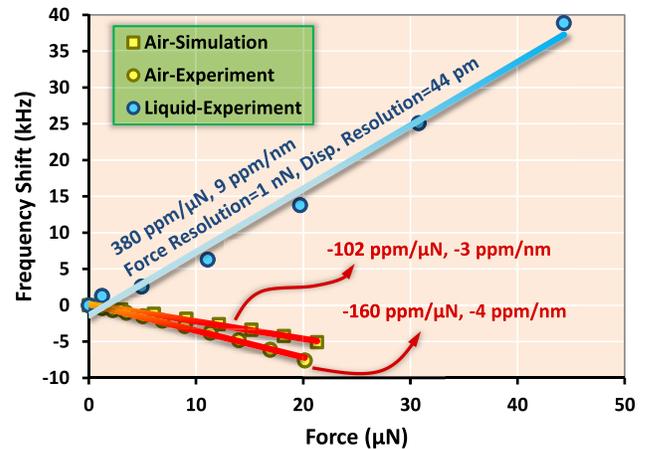


Fig. 4. Measured frequency of the resonator of Fig. 1 as a function of the applied local force.

$\sim 80$ , respectively. Xylene, which is an organic nonconductive liquid, was used to allow application of relatively large actuation voltages to the electrostatic actuator utilized here for the characterization purpose. Figure 3 shows how the frequency response of the 2.3 MHz resonator of Fig. 1 changes under constant bias current in both air and liquid, while increasing the external electrostatic force. It should be noted that, by precisely measuring the comb drive gap size after fabrication, the forces and displacements are determined based on calculations and simulations, respectively. The change in resonance frequency of the resonant force-displacement sensor of Fig. 1 as a function of the external force is illustrated in Fig. 4, showing force and displacement sensitivities of  $0.88 \text{ Hz/nN}$  ( $0.38 \text{ ppm/nN}$ ) and  $21 \text{ Hz/nm}$  ( $9 \text{ ppm/nm}$ ), respectively, in liquid. Interestingly, the results show that the shifts in liquid are  $\sim 2\times$  larger and happen in the opposite direction than in air, leading to 2-fold better force and displacement resolutions

TABLE I  
SUMMARY OF IN-LIQUID MEASUREMENT RESULTS OBTAINED FROM  
DEVICES WITH DIFFERENT DIMENSIONS (SPOTTED IN FIG. 5)

Device#	Dimensions (L×w)	Force Resolution	Stiffness (kN/m)	Displacement Resolution	FOM (nN.pn)
1	180 μm×2 μm	1 nN	0.025	40 pm	40
2	30 μm×2 μm	25 nN	12.5	2 pm	50
3	10 μm×2 μm	100 nN	500	200 fm	20

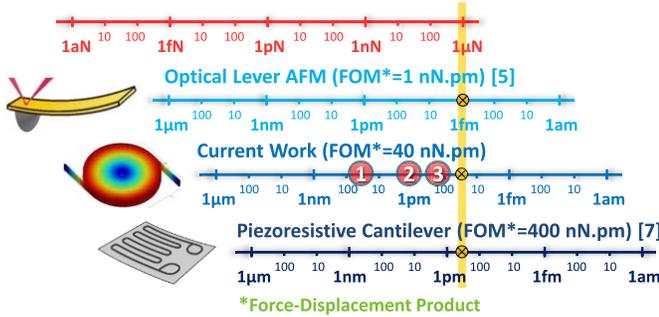


Fig. 5. Comparison of current work performance with other approaches in terms of their FOM (force-displacement product) when operating in liquid. The blue and red axes represent force and displacement resolution, respectively. A blue axis with a larger overlap with the red axis depicts a better combined force/displacement performance (lower FOM). The numbers 1 to 3 show the force and displacement sensing performance of the three resonant sensors of Table I.

( $\epsilon_r = 2.2$  has been taken into account for Xylene). This is believed to be due to the increased effect of structural deformations on viscous damping. In air, the frequency shifts are merely caused by the change in the effective structural stiffness of the device [1]. However, several other factors, possibly opposing each other, affect the operation of such devices in liquid that can contribute to the frequency shifts. For instance, a change in the damping coefficient can also cause significant shift in the frequency of such structures [10]. Since the viscosity of air is very small, damping change due to the structural deformations is essentially negligible in air, while similar deformations in liquid can cause substantial shifts in damping and in turn frequency of the devices. To validate the results, COMSOL finite element modal analysis was performed obtaining similar shifts in air (Fig. 4).

The short-term frequency stability, which determines the frequency resolution of such devices, has been measured for similar thermally actuated resonant structures using Allan variance method showing Allan deviations as low as  $1.5 \times 10^{-3}$  in liquid [11]. However, a conservative frequency measurement resolution of 1Hz has been used here turning the measured sensitivities into force and displacement resolutions of 1 nN and 40 pm in liquid, respectively. Table I summarizes the results obtained from a number of similar devices with different dimensions demonstrating displacement and force resolutions as small as 200 fm and 1 nN, respectively. It should be noted that force resolution and displacement resolution of such sensors are directly related via the structural stiffness at the point where the external force is applied, leading to a trade-off between the two, i.e. force resolution can be

improved at the expense of losing displacement resolution and vice versa. Therefore, to have a fair comparison with other atomic force measurement techniques, resolution figure-of-merit (FOM) is defined as the force resolution-displacement resolution product. Illustrating the existing tradeoff between the force and displacement resolutions within different approaches, Fig. 5 compares the performance of the proposed approach with optical lever and piezoresistive cantilevers, where a smaller FOM indicates a better combined displacement/force sensing performance. As an example, for the same force resolution of 1 μN, optical lever AFM offers the smallest displacement resolution among the three approaches which is more desirable. As it is shown, the proposed approach provides  $\sim 10\times$  and  $\sim 2\times$  better FOM in liquid (40 nN.pn) compared to typical (400 nN.pn) [7] and state-of-the-art parylene-coated (100 nN.pn) [8] piezoresistive cantilevers, respectively.

#### IV. CONCLUSIONS AND FUTURE WORK

Subatomic resolution force and displacement measurements in air and liquid were demonstrated using rotational mode disk resonant structures acting as stress/strain sensors. The measurement results were confirmed using COMSOL finite element modal analysis. The presented technique requires no external sensing element and shows  $\sim 10\times$  improvement in FOM, defined as force resolution-displacement resolution product, over typical piezoresistive cantilevers. Integration of nano-tips into such resonant MEMS devices, a new class of high resolution atomic force probes can be realized circumventing the low  $Q$  limitation of currently available AFM probes when operating in liquid media.

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