Chapter 1

Overview

1.1 Nanoelectromechanical Systems

Nanoelectromechanical systems (NEMS) are microelectromechanical devices (MEMS) scaled down to nanometer range.[1] NEMS have a lot of intriguing attributes.[2] They offer access to fundamental frequencies in the microwave range; [3] quality factor (Q) in the tens of thousands; [4] active mass in the femtogram range; force sensitivities at the attonewton level;[5,6] mass sensitivity at the level of individual molecules[7] — this list goes on. These traits translate into new prospects for a variety of important technological applications. Among them, nanomechanical resonators are rapidly being pushed to smaller sizes and higher frequencies due to their applications as Q filters and on-chip clocks.[4] The fully integrated NEMS oscillators will boast smaller size and lower power consumption and thus can potentially replace their macroscopic counterparts such as the quartz crystal oscillators and surface wave acoustic resonators.

The resonance frequency in general scales as $1/L$, where L is the scale of the resonator. As size scales are reduced and frequency is increased, the corresponding statistical fluctuations will be more pronounced and inevitably limit performance. The central question of this thesis is: as the size of the resonator becomes smaller, how stable can the resonant frequency be? The answers to this seemingly simple question form the subject of phase noise of NEMS. We will review the pioneering work before going to this subject in detail.

1.2 Brownian Motion, Nyquist-Johnson Noise, and Fluctuation-Dissipation Theorem

A microscopic particle immersed in a liquid exhibits a random type of motion. This phenomenon is called Brownian motion and reveals clearly the statistical fluctuations that occur in a system in thermal equilibrium.[8] The Einstein relation, perhaps the most important result of the study of Brownian motion, states that the diffusion constant is proportional to the frictional coefficient determined by the hydrodynamic interaction of the particle with the viscous fluid.[12] The Brownian motion serves as a prototype problem whose analysis provides considerable insight into the mechanisms responsible for the existence of fluctuations and dissipation of energy. This problem is also of great practical interest because such fluctuations constitute a background of "noise" which imposes sensitivity limits on delicate physical measurements. For example, Nyquist-Johnson noise, which originates from thermal agitation of electrical charge in a conductor,[9,10] is present at any circuitry with nonzero dissipation, and in many cases determines the noise floor of an amplifier.[11] Nyquist's theorem states that the spectral density of the thermal fluctuating voltage of any electrical impedance is always proportional to the square root of its resistive part.[13] The same arguments used to study Brownian motion and Nyquist-Johnson noise can be extended on a more abstract level to a general result of wide applicability, the fluctuation-dissipation theorem.[12-14] The fluctuation-dissipation theorem explicitly indicates how the cross-correlation functions of the fluctuating quantities are associated with the friction coefficients of the equations of motion, or equivalently, how the spectra of statistical fluctuations are related to the dissipations of the system near thermal equilibrium.

1.3 Noise in Microelectromechancial Systems and Nanoelectromechanical Systems

We now review the study of the noise of MEMS and NEMS, starting from the work in a liquid. Paul and Cross have considered the Brownian motion of NEMS cantilevers and concluded that the corresponding force sensitivities are in the range of piconewton.[15] Considering the hydrogen bond strength is ~10 pN, such sensitivities imply the possibility of using NEMS to sense biological forces at single molecule level. On the other hand, optical tweezers have recently led to quite spectacular measurements of small weak force, with the force sensitivities again limited by Brownian motion.[16] In this technique, an optical beam, focused to the diffraction limit, is employed. Functionalized dielectric beads, typically having diameters of ~1 μm, are attached to the biomolecules under study to provide a handle. In this way, direct measurements of piconetwon scale biological forces have been obtained.[18] In a more recent study, internal dynamics of DNA, yielding forces in the femtonewton range, have been observed via the two-point correlation technique.[17]

We now discuss the work on characterization the thermomechancial noise of MEMS and NEMS in vacuum. Albrecht et al. demonstrate frequency modulation detection using high Q cantilevers for enhanced force microscopy sensitivity, limited by thermomechancial noise in vacuum.[19] Similarly, using a high *Q* single crystal silicon cantilever as thin as 60 nm, T. D. Stowe et al. have achieved attonewton force sensitivity at 4.8 K in vacuum.[5] Cooling down similar devices further to millikelvin temperatures, force sensitivity at subattonewton scale has also been demonstrated.[6] Such exquisite force sensitivities have ultimately led to the detection of single electron spin using magnetic resonance force microscopy (MRFM).[20]

The observation of thermomechancial noise of high frequency NEMS has been hindered, largely due to the diminishing transducer responsivity as the dimensions are reduced into the submicron range. This can only be circumvented by delicate incorporation of the actuator, transducer, and readout amplifier, all meticulously chosen and orchestrated to minimize the noise from these extrinsic elements. For example, the piezoresistors on NEMS silicon cantilevers, which acts as transducers upon current biasing, convert the mechanical displacement into a voltage signal, which is subsequently read out by a low noise amplifier. Using such a scheme, Arlett et al. have observed the theromomechanical noise down to cryogenic temperatures for NEMS devices with resonance frequencies of ~2 MHz.[22]

Another example is the nanomechanical parametric amplifier at 17 MHz by Harrington,[23] which is similar to the one demonstrated by Rugar and Grutter using a microscale cantilever.[21] Operating in degenerate mode, a parametric modulation of the beam's effective stiffness at twice the signal frequency is produced by the application of an alternating longitudinal force to both ends of a doubly clamped beam. At highest mechanical gains, noise matching performance is achieved, resulting in the observation of thermomechanical noise squeezing at cryogenic temperatures.

Finally, we mention the recent attempt to approach the quantum limit of a nanomechancial resonator by coupling a single electron transistor (SET) with a high *Q*, 19.7 MHz nanomechanical resonator by LeHaye et al.[24] At temperatures as low as 56 millikelvin, they observe thermomechanical noise corresponding to a quantum occupation number of 58, and demonstrate the near-ideal performance of the SET as a linear amplifier. This work clearly paves the feasible way to the quantum mechanical limits of NEMS, blurring the division between quantum optics and solid state physics.[2]

1.4 Phase Noise in Microelectromechancial Systems and Nanoelectromechanical Systems

We now review work on the phase noise of NEMS and MEMS. The phase noise of MEMS resonators was first analyzed by Vig and Kim.[25] They examine how frequency stabilities of MEMS and NEMS resonators scale with dimensions. When the dimensions of a resonator becomes small, instabilities that are negligible in macroscale devices become prominent. At submicron dimensions, the temperature fluctuation noise, adsorption-desorption noise, and thermomechanical noise are likely to limit the applications of ultra small resonators. Later, Cleland and Roukes develop a self-containing formalism to treat a similar list of noise sources and estimate their impact on a doubly clamped beam of single crystal silicon with a resonance frequency of 1 GHz.[26] Their calculation, however, does not agree with Vig and Kim's work in terms of the magnitude of the impact of the noise, as well as the method of analysis of some of the noise sources, in particular, that of the effect of temperature fluctuations. In analyzing the temperature fluctuation noise, they consider a more realistic thermal circuit by dividing the device into sections, and show that the resulting Allan variance is of the same magnitude as that due to thermomechanical noise for the model resonator with Q of 10^4. This apparently contradicts the excessive temperature fluctuations predicted by Vig and Kim.[25] Moreover, they conclude that the noise performance, limited by the fundamental noise processes, can be comparable with their macroscale counterparts, the oven stabilized quartz crystal oscillators. By consolidating these studies, we first introduce the subject of phase noise in chapter 2, and then present the theory of the phase noise mechanisms affecting NEMS in chapter 3.

Except for the aforementioned theoretical works, very little experimental data are available for evaluating whether the calculated noise performance can be achieved. More systematic approaches, measuring the performance of high Q resonators operating in phase-locked loops, with controlled variations in temperature, environment, and materials, need to be followed. As an initial step into these efforts, we describe the implementations of phase-locked loops based on NEMS devices in chapter 4. We also report the observation of adsorption-desorption noise arising from xenon atoms adsorbed on the device surface. Our measurement results are in excellent agreement with the proposed idea gas model. More generally, our approach represents a canonical example on how to study the frequency stabilities arising from a particular noise process of interest.

1.5 Mass Sensing Based on Microelectromechanical Systems and Nanoelectromechancial Systems

We now review a separate, but closely related front: the inertial mass sensing based on MEMS and NEMS. Today mechanically based sensors are ubiquitous, having a long history of important applications in many diverse fields of science and technology. Among the most responsive sensors are those based on the acoustic vibratory modes of crystals,[27,28] thin films,[29] and more recently, MEMS[30,31] and NEMS.[7,32,] Three attributes of these devices establish their mass sensitivity: effective vibratory mass, quality factor, and resonant frequency. The miniscule mass, high Q, and high resonant frequency of NEMS provide them with unprecedented potential for mass sensing. Femtogram mass sensing using NEMS cantilevers has been demonstrated by Lavrik and Datskos by photothermally exciting silicon cantilevers in the range of 1 to 10 MHz and measuring a mass change of 5.5 fg upon chemisorption of 11-mercaptoundecanoic acid.[32] Ekinci and Roukes achieve attogram mass sensing by exposing NEMS devices with Au atomic flux and tracking the resulting frequency shift in a phase-locked loop.[33] Motivated by these experiments, we start to examine theoretically the ultimate limits of inertial mass sensing based upon NEMS devices as a result of fundamental noise processes.[7] We present the resulting theoretical analysis in chapter 3. The conclusion is quite compelling: it indicates that NEMS devices can directly “weigh” individual intact, electrically neutral, molecules with single Dalton sensitivities.

As an initial step toward this goal, we present our mass sensing experiments at zeptogram scale in chapter 4. This is demonstrated by depositing xenon atoms and nitrogen molecules on the NEMS device, and tracking the resulting frequency shift in high precision phase-locked loop. But more importantly, the agreement of our experimental results with the theory justifies our formalism and validates its use to delineate, for the first time, the feasible pathway into single Dalton sensitivity.

1.6 Organization

To help the reader understand this work in a more coherent and clear way, this thesis is organized in the following way:

Chapter 2 introduces the subject of phase noise and serves as the mathematical foundation of this work. We first describe how phase fluctuations of an oscillator convert to the noise sideband of the carrier. We then define the phase noise, the frequency noise, and Allan deviation, emphasizing their relationship with each other. As an example, Leeson's model is described and used to analyze the thermal noise of an ideal linear LC oscillator.

Chapter 3 discusses the phase noise mechanism of the NEMS resonators. We first examine fundamental noise processes, including thermomechancial noise, momentum exchange noise, adsorption-desorption noise, diffusion noise, and temperature fluctuation noise. We also discuss nonfundamental noise processes arising from the Nyquist-Johnson noise of the transducer amplifier implementations. For each noise process presented here, we give expressions for the phase noise spectra and Allan deviation and then translate them into the corresponding minimum measurable frequency shift and mass sensitivity in light of their importance in sensing applications.

Chapter 4 presents the experimental measurement of the phase noise of NEMS. First, we first analyze the control servo behavior of the phase-locked loops and give the detailed implementations together with their noise performance. The achieved noise performance is compared to the local oscillator (LO) requirements of chip scale atomic clocks (CSAC) to evaluate the viability of NEMS based oscillators for this application. Finally, we investigate the diffusion noise arising from adsorbed xenon atoms by putting a very high frequency NEMS into the phase-locked loop and measuring the frequency noise spectra and Allan deviation.

Chapter 5 shows very high frequency NEMS that provide a profound sensitivity increase for inertial mass sensing into zeptogram scale. We demonstrate *real time*, *in situ* mass detection of sequential pulses of ~100 zg nitrogen molecules by tracking resulting frequency shift. Measurement and analysis from our experiments demonstrate mass sensitivities at the level of ~7 zg, the mass of an individual 4 kDa molecule, or ~30 xenon atoms.

Chapter 6 describes a surface nanomachining process that involves electron beam lithography, followed by dry anisotropic and selective electron cyclotron resonance plasma etching steps. Measurements on a representative family of the resulting devices demonstrate that, for a given geometry, nanometer-scale SiC resonators are capable of yielding substantially higher frequencies than GaAs and Si resonators.

Chapter 7 describes a broadband radio frequency balanced bridge technique for electronic detection of displacement in NEMS. The effectiveness of the technique is demonstrated by detecting the minute electromechanical impedances of NEMS embedded in large electrical impedances at very high frequencies.

References

1. M. L. Roukes Plenty of room indeed. *Sci. Am.* **285**, 48 (2001).

2. M. L. Roukes Nanoelectromechanical Systems. *Technical Digest, Solid State Sensor and Actuator Workshop, Hilton Head Island, South Carolina (June4–8, 2000)* 367–376 (2000).

3. X. M. H. Huang, C. A. Zorman, M. Mehregany, and M. L. Roukes Nanodevices motion at microwave frequencies. *Nature* **421**, 496 (2003).

4. A. N. Cleland and M. L. Roukes Fabrication of high frequency nanometer scale mechanical resonators from bulk Si crystals *Appl. Phys. Lett.* **69**, 2653 (1996).

5. T. D. Stowe, K. Yasumura, T.W. Kenny, D. Botkin, K. Wago, and D. Rugar Attonewton force detection using ultrathin silicon cantilevers. *Appl. Phys. Lett.* **71**, 288 (1997).

6. H. J. Mamin and D. Rugar Sub-attonewton force detection at milikelvins. *Appl. Phys. Lett.* **79**, 3358 (2001).

7. K. L. Ekinci, Y. T. Yang, and M. L. Roukes Ultimate limits to inertial mass sensing based upon Nanoelectomechancial systems *J. Appl. Phys*. **95**, 2682 (2004).

8. A. Einstein *Investigation on the Theory of the Brownian Movement* (New York, Dover, 1956).

9. H. Nyquist Thermal agitation of electrical charge in conductors. *Phys. Rev.* **32**, 110 (1928).

10. J. B. Johnson Thermal agitation of electricity in conductors. *Phys. Rev.* **32**, 97 (1928).

11. P. R. Grey and R. G. Meyer *Analysis and Design of Analog Integrated Circuits* (New York, John Wiley & Sons, 1984).

12. P. M. Chaikin and T. C. Lubensky *Principles of Condensed Matter Physics* (New York, Cambridge University Press, 1995).

13. F. Reif *Fundamentals of Statistical and Thermal Physics* (Singapore, McGraw-Hill, 1996).

14. L. D. Landau and E. M. Lifshitz *Statistical Physics* (London, Oxford, 1980).

15. M. R. Paul and M. C. Cross Stochastic dynamics of nanoscale mechanical oscillators immersed in a viscous fluid. *Phys. Rev. Lett.* **92**, 235502-1, (2004).

16. J. C. Crocker Measurement of the hydrodynamic corrections to the Brownian motion of two colloidal spheres. *J. Chem. Phys.* **106**, 2837 (1997).

17. J. C. Meiners and S. R. Quake Femtonewton force spectroscopy of single extended DNA molecules. *Phys. Rev. Lett.* **84**, 5014 (2000).

18. K. Visscher, M. J. Schnitzer and S. M. Block Kinesin motors studied an optical force clamp. *Biophysical Journ.* **74**, A49 (1998).

19. T. R. Albrecht, P. Grutter, D. Horne, and D. Rugar Frequency modulation detection using high Q cantilever for enhanced force microscopy sensitivity *J. Appl. Phys.* **69**, 668 (1991).

20. D. Rugar, R. Budakian, H. J. Mamin, and B. W. Chui Single spin detection by magnetic resonance force microscope. *Nature* **430**, 329 (2004).

21. D. Rugar and P. Grutter Mechanical parametric amplification and thermomechanical noise squeezing. *Phys Rev. Lett.* **67**, 699 (1991).

22. J. L. Arlett, J. R. Maloney, B. Gudlewski, M. Muluneh, and M. L. Roukes Self-sensing micro- and nanocantilevers with attonewton-scale force resolution. *Nano Lett.* **6**, 1001, (2006).

23. D. A. Harrington Physics and applications of nanoelectromechanical systems (NEMS). PhD thesis, California Institute of Technology (2003).

24. M. D. LaHaye, O. Buu, B. Camarota, and K. C. Schwab Approaching the quantum limit of a nanomechanical resonator. *Science* **304**, 74 (2004).

25. J. Vig and Y. Kim Noise in microelectromechanical system resonators *IEEE Trans. on Ultrasonics, Ferroelectronics and Frequency Control* **46**, 1558 (1999).

26. A. N. Cleland and M. L. Roukes Noise processes in nanomechanical resonators. *J. Appl. Phys.* **92**, 2758 (2002).

27. D. S. Ballantine et al. *Acoustic Wave Sensors* (San Diego, Academic Press, 1997).

28. C. Lu *Application of Piezoelectric Quartz Crystal Microbalance* (London, Elsevier, 1984).

29. M. Thompson and D. C. Stone *Surface-Launched Acoustic Wave Sensors: Chemical Sensing and Thin Film Characterization* (New York, John Wiley and Sons, 1997).

30. J. Thundat, E. A. Wachter, S. L. Sharp, and R. J. Warmack Detection of mercury vapor using resonating microcantilevers. *Appl. Phys. Lett.* **66**, 1695-1697(1995).

31. Z. J. Davis, G. Abadal, O. Kuhn, O. Hansen, F. Grey, and A. Boisen Fabrication and characterization of nanoresonting devices for mass detection. *J. Vac. Sci. Technol.* **B 18**, 612-616(2000).

32. N. V. Lavrik and P. G. Datskos Femtogram mass detection using photothermally actuated nanomechanical resonators. *Appl. Phys. Lett* **82**, 2697 (2003).