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## **Viscoelastic Coupling of Nanoelectromechanical Resonators**

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## **Abstract**

This report summarizes work to date on a new collaboration between Sandia National Laboratories and the California Institute of Technology (Caltech) to utilize nanoelectromechanical resonators designed at Caltech as platforms to measure the mechanical properties of polymeric materials at length scales on the order of 10-50 nm. Caltech has succeeded in reproducibly building cantilever resonators having major dimensions on the order of 2-5 microns. These devices are fabricated in pairs, with free ends separated by reproducible gaps having dimensions on the order of 10-50 nm. By controlled placement of materials that bridge the very small gap between resonators, the mechanical devices become coupled through the test material, and the transmission of energy between the devices can be monitored. This should allow for measurements of viscoelastic properties of polymeric materials at high frequency over short distances. Our work to date has been directed toward establishing this measurement capability at Sandia.

## **ACKNOWLEDGMENTS**

We gratefully acknowledge collaboration with Dr. Edward B. Myers, Dr. Xinchang Zhang, and Professor Michael Roukes of the Departments of Physics and Applied Physics at the California Institute of Technology. In addition, we thank Mr. Ric Watson of eDT, Inc. (Albuquerque, NM) for his assistance in fabrication of radiofrequency drive and sensing circuitry for control of nanoelectromechanical cantilever devices.

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## Nomenclature

DOE	Department of Energy
cm	centimeters
Hz	Hertz, unit of frequency in cycles per second
MEMS	Microelectromechanical systems
mm	millimeters
NEMS	Nanoelectromechanical systems
nm	nanometer
RF	radiofrequency
SNL	Sandia National Laboratories

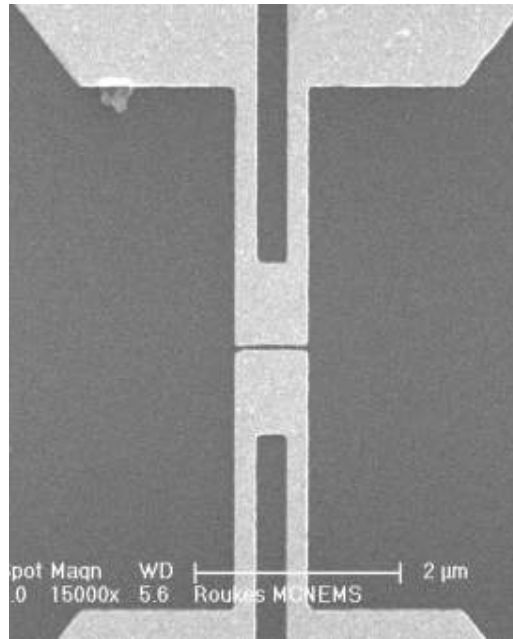
# 1. INTRODUCTION

The mechanical properties of polymeric materials are usually key considerations in the application of those materials to engineering problems. Accordingly, an extensive literature exists regarding the measurement of mechanical properties of polymers at small deformations [1]. These measurements typically require macroscopic samples of the materials under investigation, with sample dimensions on the order of mm to cm. However, as microelectromechanical (MEMS) and nanoelectromechanical (NEMS) devices become more prominent in engineered systems, mechanical dimensions of thin film materials can become much smaller than the typical dimensions used to ascertain bulk material properties. For NEMS devices, material dimensions on the order of 10-100 nm are becoming more and more common. At such dimensions the assumptions of smoothly varying (continuous, differentiable) mechanical properties of materials can break down, calling into question the extrapolation of material properties (e.g., elastic modulus, loss modulus) measured for bulk samples to this much smaller length scale. Accordingly, in recent years efforts have increased in both direct measurements [2,3] and modeling [4] of materials deformation and fracture at 10-100 nm dimensions. However, a potentially serious problem exists in the use of nanoindentation-based materials property measurements to predict the behavior of NEMS devices. Typical nanoindentation strain rates are relatively slow. Transformation of typical nanoindentation displacement rates per unit time results in measurements over a frequency range on the order of 1-100 Hz [3]. In contrast, NEMS devices often operate in the MHz or even GHz frequency range [5]. Thus, the results of nanoindentation-based materials studies would need to be extrapolated over 4-7 orders of magnitude to be applied to some NEMS devices.

We have begun collaboration with researchers at the California Institute of Technology in order to attempt to address this discrepancy, based on an idea first proposed by Dr. Edward Myers of Caltech. Our approach relies on the use of coupled pairs of NEMS cantilever resonators as platforms for the direct measurement of mechanical energy transmission through suspended films of polymeric materials. The cantilevers are fabricated by electron beam lithography in SiC or SiN layers with typical in plane dimensions of 2.5 microns length x 0.7 micron width. The cantilevers are manufactured in pairs that are arranged “end-to-end”, such that the free ends of the cantilevers are separated by a narrow gap (10-50 nm). A freestanding polymer membrane is formed across the gap by dropcoating or spraycoating polymer solutions onto the NEMS devices. Solvent evaporation results in formation of a polymer “bridge” between the two NEMS resonators. By driving one cantilever across a range of frequencies and measuring the amplitude and phase of displacement of the other NEMS cantilever, it should be possible for us to measure the elastic coupling and viscous energy loss in the polymer. Thus the coupled NEMS devices will form a novel measurement platform for the direct measurement of material properties, at nanometer dimensions and at excitation frequencies relevant to the operation of NEMS devices.

## 2. PROOF OF CONCEPT MEASUREMENTS AT CALTECH

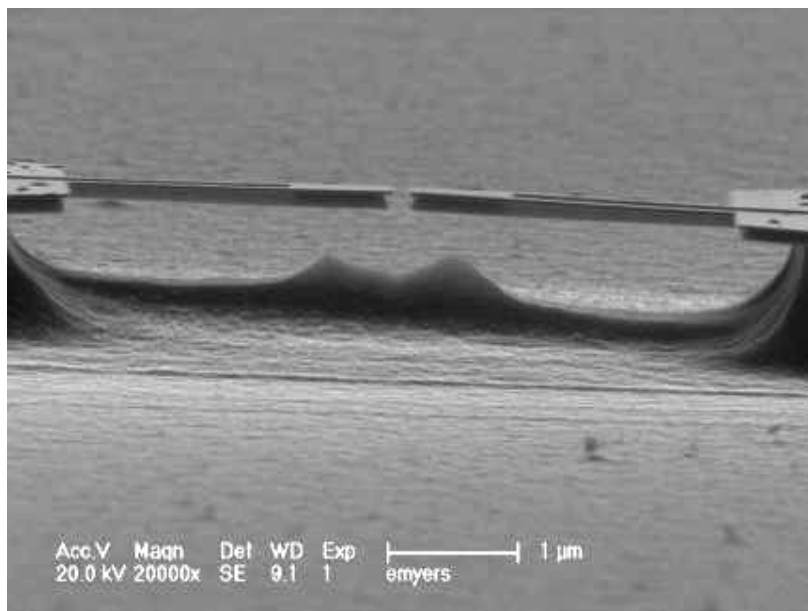
The NEMS sensors are fabricated from 100-nm-thick low-stress silicon nitride films grown on bulk silicon. Electron-beam lithography, combined with gold deposition (30 nm thick) and liftoff, are used to define the resonator pattern, a two-legged cantilever. The gold pattern serves both as a piezoresistive displacement detector for the resonator as well as an etch mask for the subsequent release step. After liftoff, a two-step plasma etch is performed to first anisotropically etch through the nitride, and then to isotropically etch the silicon to release the NEMS structure. A “generation 1” test device fabricated by Dr. Ed Myers and coworkers at Caltech is displayed in Figure 1.



**Figure 1. Paired NEMS cantilevers fabricated at Caltech.**

The paired cantilevers in Figure 1 are 2.5 microns long and 0.7 microns wide. The gap between the ends is approximately 50 nm. An oblique view of this cantilever pair after release etching is shown in Figure 2. These cantilevers can be driven thermoelastically [6]. The heat capacity of these tiny devices is sufficiently small, and the thermal conductance to the substrate sufficiently large, that driving a heating current through the gold coating at high frequency results in expansion and contraction of the cantilevers with little phase lag. The coupling of this thermal expansion to the fundamental out of plane bending mode of the cantilever serves to drive the device, and by sweeping the drive frequency the resonance of the cantilevers can be detected. In practice, the motion of the cantilevers is measured by piezoresistive downmixing, where the difference between the cantilever displacement frequency and a discrete reference frequency is measured using a lock-in amplifier [7].

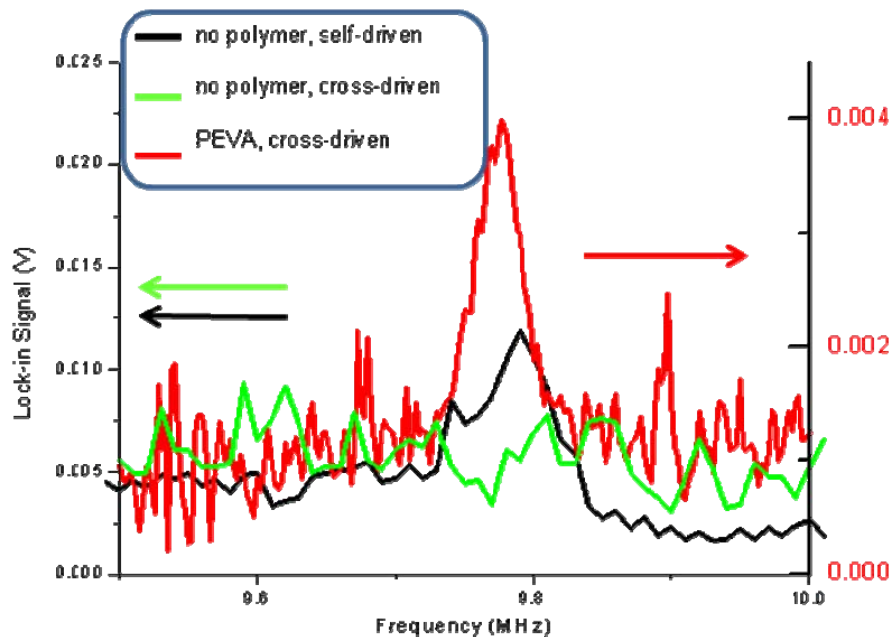




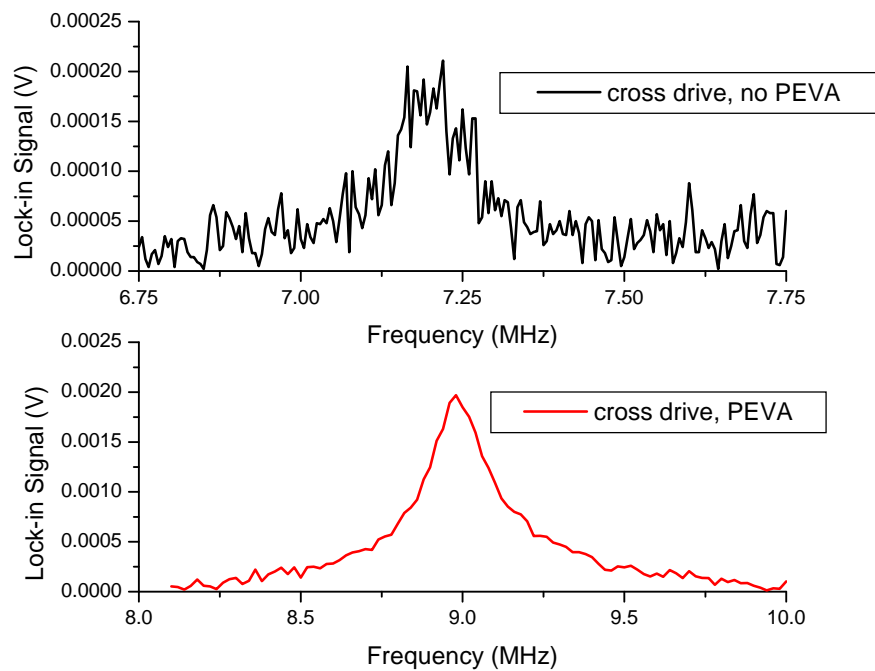
**Figure 2. Oblique view of paired NEMS cantilevers after release etch.**

Experiments were performed by Caltech researchers to demonstrate proof-of-concept of the mechanical coupling of these cantilevers by a polymer film suspended in the gap. A copolymer of polyethylene containing 18% vinyl alcohol monomer (PEVA) was solution-deposited on the paired devices displayed in Figures 1 and 2 by dropcoating. The frequency and amplitude of the resonances of both cantilevers were then measured in “self-drive” and “cross-drive” modes. In self-drive mode the thermoelastic driving signal was applied to one cantilever of the pair, and the response of that cantilever was measured. In cross-drive mode the thermoelastic driving signal was applied to one cantilever of the pair, and the response of the other cantilever was measured. Some example results are displayed in Figure 3, where the amplitude of the fundamental vibration mode of the cantilevers is shown as a function of driving frequency for both self-drive and cross-drive operation. The amplitude of the cross-drive signal is undetectable under these conditions for the uncoupled devices, while a peak is observed in the cross-drive signal for the PEVA-coupled devices. This demonstrates the proof-of-concept coupling of the devices through the polymer film.

The signal to noise ratio is low for the resonance data of Figure 3. A pair of NEMS resonators with a narrower gap ( $\sim 15$  nm) was fabricated at Caltech by gold coating a fine wire across the gap prior to the release etch. During deposition the wire exhibited gaps due to grain coarsening of the deposited Au. After release etch gaps between the paired cantilevers of  $\sim 15$  nm were observed by scanning electron microscopy (not shown). A pair of these “generation 2” devices was evaluated at Caltech in cross-drive mode before and after drop coating with PEVA. The results are displayed in Figure 4. The signal to noise ratio is superior in these measurements to that measured for the generation 1 resonators. With no PEVA, there is still a detectable cross-drive signal, which may be due to electrostatic coupling of the cantilevers. After PEVA deposition the amplitude of the cross-drive signal increased by approximately a factor of 10. In addition, the resonance frequency shifted by approximately 25%. Our goal is to look for such shifts in amplitude, frequency, and phase in cross-drive measurements and to attempt to relate these measurements to the mechanical properties of the bridging materials.



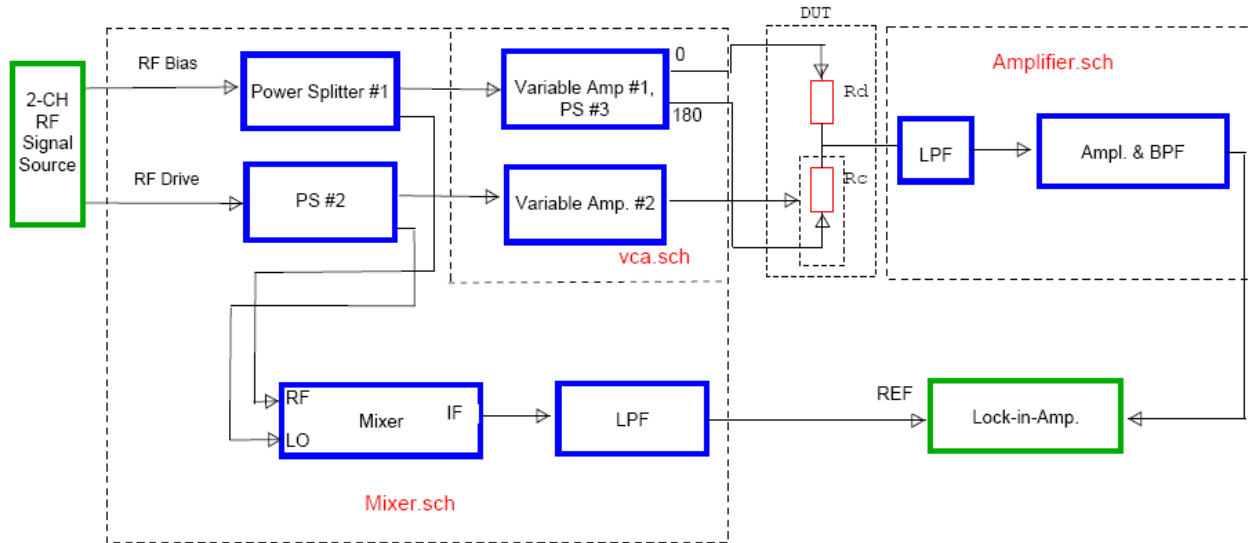
**Figure 3. Measurements at Caltech of cross-drive vs. self-drive resonance of paired cantilevers separated by a 50 nm gap. The gap was bridged by a film of polyethylene vinyl alcohol (PEVA).**



**Figure 4. Resonances of 15nm gap NEMS with and without PEVA film.**

### 3. NEMS MEASUREMENT CAPABILITY AT SANDIA

The goal of this late-start LDRD project is to produce a capability at Sandia for the measurement of frequency and amplitude of NEMS resonator pairs in the cross-drive mode. Accordingly, the primary task has been the duplication of specialized drive and sensing electronics for the thermoelastic excitation and piezoresistive measurement of NEMS oscillators. A block diagram of the required system is shown in Figure 5. Details regarding the principle of circuit operation are discussed in References 6 and 7.



**Figure 5. Block diagram of NEMS drive and sensing circuits.**

The block components shown in green in Figure 5 are commercial components that can be controlled by PCI interface on a personal computer running LabView™ software. The 2-channel RF signal source is an Acquitex™ Synth-300 direct digital synthesizer. The lock-in amplifier is a Femto Messtechnik GmbH single-board dual phase amplifier, LIA-BVD-150-L. All other functions in the block diagram including DC power, RF amplification, RF mixing, and low-frequency amplification are performed by a custom circuit board design provided by Dr. Xinchang Zhang of the California Institute of Technology. Two of these custom circuit boards have been built by Mr. Ric Watson of eDT, Inc. (Albuquerque, NM), and have now been functionally tested by Sandia personnel and by Dr. Zhang. In addition, a PCI expansion chassis and the commercial synthesizer and lock-in components have been obtained and tested at Sandia. Thus, the capability for measuring coupled NEMS oscillator resonances is now in place at Sandia National Laboratories.

## 4. CONCLUSIONS

Through collaborations with researchers at the California Institute of Technology, the capability of making cross-drive measurements of coupled nanoelectromechanical resonators has been reproduced at Sandia National Laboratories. The purpose of establishing this capability is to allow for testing of viscoelastic mechanical properties of polymeric materials at the 10-50 nm length scale, and at MHz frequencies relevant to the operation of novel NEMS devices. Nanoindentation based methods can also measure viscoelastic properties at this length scale, but the displacement speeds of nanoindenters limit such measurements to much lower frequencies (1-100 Hz). Our initial experiments will concentrate on the measurement of mechanical properties of SU8 photoresist at high frequency. This will serve as a test case for the new measurement apparatus, since bulk material properties and nanoindentation properties of SU8 at low frequency have been documented in the literature [8].

## 5. REFERENCES

1. Ferdinand Rodriguez, *Principles of Polymer Systems*, 3<sup>rd</sup> edition, Hemisphere Publishing Corporation (New York) 1989.
2. Yu. I. Golovin, “Nanoindentation and Mechanical Properties of Solids in Submicrovolumes, Thin Near-Surface Layers, and Films: A Review”, *Physics of the Solid State* **50**(12), 2205-2236, 2008.
3. J.E. Houston, “A Local-Probe Analysis of the Rheology of a “Solid Liquid”, *J. Polymer Sci.* **B43**(21), 2993-2999 (2005).
4. M.L. Parks, R.B. Lehoucq, S. Plimpton, and S. Silling, “Implementing peridynamics within a molecular dynamics code”, *Computer Physics Communications* **179**, 777-783 (2008).
5. Mo Li, H.X. Tang, and M.L. Roukes, “Ultra-sensitive NEMS-based cantilevers for sensing, scanned probe and very high frequency applications”, *Nature Nanotechnology* **2**, 114-120, 2007.
6. I. Bargatin, I. Kozinsky, and M.L. Roukes, “Efficient electrothermal actuation of multiple modes for high-frequency nanoelectromechanical resonators”, *Applied Physics Letters* **90**, 093116 (2007).
7. I. Bargatin, E.B. Myers, J. Arlett, B. Gudlowski, and M.L. Roukes, “Sensitive detection of nanomechanical motion using piezoresistive signal downmixing”, *Applied Physics Letters* **86**, 133109 (2005).
8. K.I. Schiffmann and C. Brill, “Testing the viscoelastic properties of SU8 photo resist thin films at different stages of processing by nanoindentation creep and stress relaxation”, *Int. J. Mat. Res.* **98**(5) 397-403 (2007).

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