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978-1-107-41229-3 - Materials Science of Microelectromechanical Systems (MEMS) Devices III

Edited by Harold Kahn, Maarten de Boer, Michael Judy and S. Mark Spearing

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## **Metrology**

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Mat. Res. Soc. Symp. Proc. Vol. 657 © 2001 Materials Research Society

### Temperature-Dependent Internal Friction in Silicon Nanoelectromechanical Systems

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#### ABSTRACT

The mechanical properties of micro- and nanomechanical systems are of interest from both fundamental and technological standpoints. High-frequency mechanical resonators presenting high quality factors are of interest for the development of sensitive force detecting devices, and highly efficient RF electromechanical filters and oscillators. Internal losses are the combination of both extrinsic and intrinsic issues that must be well understood for the optimization of resonator quality, and for the experimental access to fundamental nanoscopic mechanical phenomena. The temperature dependent internal friction in 1-10 MHz paddle oscillators is reported. Quality factors as high as 1000 and 2500 are observed at room temperature in metallized and non-metallized devices, respectively. Internal friction peaks are observed in all devices in the  $T = 160\text{--}180\text{ K}$  range. The position of those peaks is consistent with the Debye relaxation of previously reported surface and near-surface phenomena.

#### INTRODUCTION

Nanometer scale science and engineering is a broad and interdisciplinary area that has been growing explosively worldwide in the past few years. It has the potential of revolutionizing materials and product manufacturing, and the range and nature of functionalities that can be accessed. Microelectromechanical systems (MEMS) have already revolutionized the microelectronics industry by providing a new range of *integrative* functionalities to booming fields such as biotechnology, communications, and safety devices. Advances in nanomachining, actuation, and mechanical characterization have recently allowed the fabrication and operation of freestanding objects in silicon and other materials, with thicknesses and lateral dimensions down to about 20 nanometers. [1] Such reduction towards *nanoelectromechanical systems* (NEMS) would bestow highly fruitful new applications such as nanobiotechnology, [1] attonewton detection, [2,3] and radio frequency (rf)-range operation. [4] The development of rf-range nanomechanical resonators are particular commercial interest to wireless systems by offering an *integrative* alternative to surface acoustic wave (SAW) technology. [4] Issues ranging from fundamental materials science to manufacturability hinder such deployment. Energy dissipation is already known to increase with reduced dimensions, [5,6] severely limiting the quality of nanometer scale devices. Such dissipative processes must be better understood in rf-range resonators in order to deploy such devices in wireless applications that demand high spectral purity.

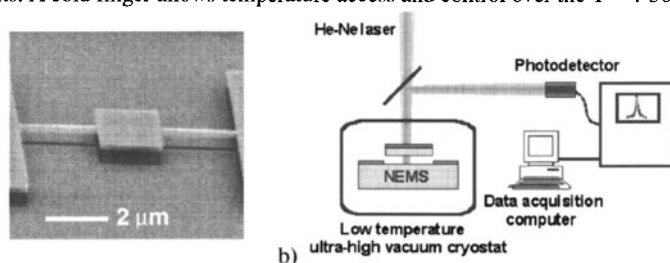
The mechanisms leading to energy dissipation are usually governed by an activation energy that will induce Debye relaxation peaks in the temperature dependence of internal friction. Analysis of such peaks would offer useful information on the relationship between nanostructural issues such as crystalline orientation, grain boundaries, surface effects and energy dissipation in NEMS. Such nanostructural understanding of energy dissipation in NEMS is not only driven by device-oriented motivations. It will also guide the design and understanding of bulk nanostructured materials.

We have recently reported the fabrication and electrostatic operation of nanomechanical beams as thin as 50 nm and frequencies as high as 380 MHz. [7] We have also reported the fabrication and non-linear dynamics of paddle oscillators operating in the 1-10 MHz range. [8,9] Here we report preliminary results on the temperature dependent behavior of these rf nanomechanical resonators. We observe internal friction peaks in the  $T = 160\text{ K} - 190\text{ K}$  range, which we associate with surface and near-surface phenomena previously reported in larger kilohertz range cantilevers. [5,6]

## EXPERIMENTAL

The RF excitation and interferometric detection of nanomechanical nanostructures has been described elsewhere [10]. Paddle resonators are produced using electron beam lithography on silicon-on-insulator wafers consisting of a 400 nm-thick oxide buried underneath 200 nm of single-crystal (100) silicon. Unless noted otherwise, the device consists of a 200 nm thick, 2  $\mu\text{m}$ -long, and 2-5  $\mu\text{m}$  wide paddle supported by 150-200 nm wide beams, as measured by scanning electron microscopy (Figure 1a). Given the relative rigidity of the paddle, most of the internal phenomena are expected to occur through the flexion and/or torsion of the supporting beams.

Devices are operated in a cryostat pumped down to the  $10^{-5}$  Torr range (Figure 1b). The tracking output of a Hewlett Packard ESA-L1500A spectrum analyzer provides a radio frequency (rf) actuation bias between the structures and the grounded substrate. A He-Ne laser is focused onto the paddle using a 0.35 NA microscope objective. The motion of the paddle modulates the reflected signal through interferometric effects. Modulation of the reflected laser beam is detected by a New Focus 1601 AC coupled photodetector, whose output is fed to the input of the spectrum analyzer. This technique offers a sub-nanometer sensitivity to vertical displacements. A cold finger allows temperature access and control over the  $T = 4\text{--}300\text{ K}$  range.



**Figure 1.** a) Scanning electron micrograph of a nanofabricated single-stage paddle oscillator. b) Low-temperature resonance assaying setup.

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The resonant response of a structure is acquired by sweeping the drive frequency. Assuming a Lorentzian shape of the peak intensity, the quality,  $Q$ , is closely approximated from the width of the resonance using the relation:

$$Q = \frac{f_0}{\Delta f_{FWHM}} \quad (1)$$

where  $f_0$  is the center of the resonance response, and  $\Delta f_{FWHM}$  is the full-width at half-maximum of its intensity. Defect motion is governed by an activation energy that will induce Debye relaxation peaks in the temperature dependence of internal friction. The position of these peaks is given by:

$$\frac{1}{Q}(T) \propto \frac{\omega\tau(T)}{1 + (\omega\tau(T))^2} \quad (2)$$

where  $\omega$  is the frequency of oscillation, and  $\tau(T)$  is the thermally activated motion lifetime of the defect:

$$\tau(T) = \tau_0 \exp\left(\frac{E_a}{k_B T}\right) \quad (3)$$

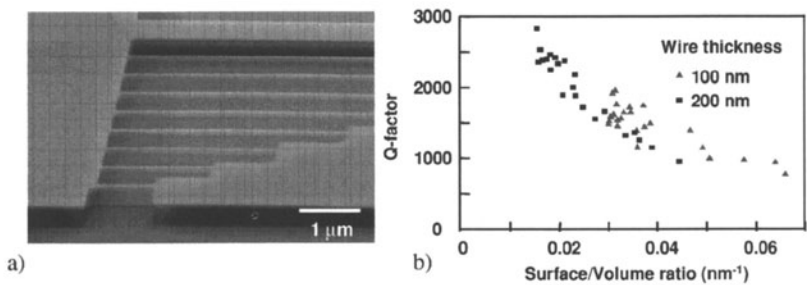
where  $E_a$  is the activation energy. Internal friction will therefore be maximum at the temperature at which  $\omega\tau(T)=1$ . Analysis of such peaks at various resonant frequencies  $\omega$  therefore offers useful information on defect thermodynamics and their impact on mechanical properties.

Our earlier devices included thin overlayers of Cr (3 nm) and Au (17 nm) evaporated on the entire structure to insure adequate electrostatic actuation. We have also successfully actuated paddle oscillators without any metal overlayers by placing a bonding wire directly on the top silicon layer within a few tens of microns of the device, and similarly bonding the grounding wire on the bottom surface also in close proximity of the device. We will therefore comparatively discuss the impact of such metal overlayers on the performance of the paddles.

## RESULTS AND DISCUSSION

### Internal friction in nanofabricated silicon beams

We have previously reported the machining and assaying of silicon beams with lateral dimensions as small as 45 nm, and resonant frequencies as high as 380 MHz. [7] Room-temperature studies of those beams revealed a strong surface-to-volume dependence of the internal friction. These early results suggested that some surface- or near-surface related phenomenon dominated energy dissipation in devices of such dimensions (Figure 2).

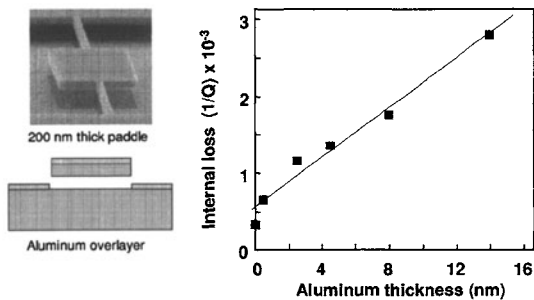


**Figure 2.** a) Scanning electron micrograph of nanofabricated silicon beams. b) Dependence of quality factor on surface-to-volume ratio. Beams are 1–8 μm-long, 100 and 200 nm thick, with lateral widths varying from 45 nm to 200 nm.

**Room-temperature internal friction in metallized paddle resonators**

Turning to the paddle resonators shown in Figure 1 a), we have identified two modes of oscillation attributed to the flexural and torsional motion of the supporting beams, respectively [8]. The resonant frequencies of these modes of motion range between 1 and 10 MHz. They are sufficiently decoupled to allow their independent excitation through the application of the proper actuation frequency.

Many MEMS employ a thin metal overlayer to ensure adequate conductivity of the structures. Given that metals have much higher internal friction than Si, they are expected to significantly degrade the quality of the thin resonators described here. Figure 3 shows the internal loss of 200 nm thick resonators completely covered with an increasingly thick layer of aluminum. The metal is a source of additional internal friction, which grows nearly linearly with the thickness of the metal layer. A sixfold increase of internal dissipation is recorded for an aluminum thickness of 14 nm.

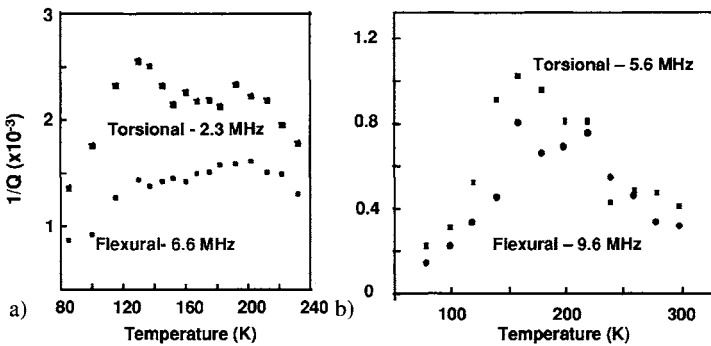


**Figure 3.** Effect of aluminum overlayer on energy dissipation in nanofabricated paddle oscillators. Torsional motion at room-temperature.

### Temperature-dependent internal friction in paddle resonators

Figures 4a) and 4b) shows the temperature dependence of the internal friction for the two modes of motion of Au/Cr metallized non-metallized devices, respectively. The reduction of the background in the non-metallized device indicates that the metal film significantly contributes to the total internal friction in that temperature range. While dominant at room-temperature (Figure 3), the contribution from the metal layer could possibly peak at much higher temperatures, as expected from bulk polycrystalline metals. [11]

All data from the metallized and non-metallized paddles show a peak structure centered at  $T = 160\text{--}180\text{ K}$ . The sustained presence of this peak suggests that the metal overlayer is not responsible for the specific dissipative mechanism peaking in that range. A similar peak has been observed at  $T = 135\text{ K}$  in larger kilohertz range microcantilevers, and has been attributed to surface or near-surface related phenomena such as damage or presence of oxide. [5,6] The peaks observed in our megahertz-range devices could potentially be related to similar phenomena, as a shift from  $T = 120\text{--}140\text{ K}$  at  $2\text{--}10\text{ kHz}$  to  $T = 160\text{--}180\text{ K}$  at  $5\text{--}7\text{ MHz}$  would be consistent with a Debye relaxation behavior dictated by an activation energy of  $E_a = 0.25\text{--}0.5\text{ eV}$ .



**Figure 4.** Temperature dependence of the internal losses for the two modes of motion of (a) Au/Cr metallized and (b) non-metallized nanofabricated silicon oscillators.

Furthermore, the dependence quality in surface-to-volume ratio observed in the single beams would be consistent with the dominance of dissipative mechanisms originating from such near-surface phenomena. Finally, the characterization of both modes of motion of these single-stage paddles consistently suggested a material 50 % softer than expected from bulk silicon. [8] This discrepancy would also support a substantial departure from bulk silicon properties within the volume of the support beams. Control in-situ annealing experiments will provide important insights for the exact identification of this internal friction process, and its relationship with the phenomena previously reported in kilohertz range devices. Furthermore, nanostructural inspection using high-resolution transmission electron microscopy would directly identify any damaged or oxidized layers that may be at the root of the observed behavior, and help elucidate the exact mechanism through which they affect device performance.

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## CONCLUSIONS

We have reported the temperature-dependent mechanical behavior of paddle oscillators with nanometer-scale supporting beams. A temperature dependent frequency shift has been observed. Low-temperature studies of internal friction at 5-7 MHz have revealed internal friction peaks centered in the  $T = 160\text{--}180\text{ K}$  range that would be consistent with near-surface phenomena previously reported in larger devices. A surface or near-surface modification of the silicon properties is also consistent with previously reported analysis of our devices. Further investigation will allow a thorough understanding of the various extrinsic, intrinsic, and fundamental processes leading to internal losses at such scales. It will enhance the quality of such RF structures, allow the development of high-quality resonators for technological applications, and provide access to fundamental studies of surface effects and mesoscopic internal friction.

## ACKNOWLEDGMENTS

Fabrication of devices was performed at the Cornell Nanofabrication Facility. This work was funded by the National Science Foundation through grants to the Cornell Center of Materials Research and the Cornell Nanofabrication Facility.

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### Resonating Microelectromechanical Structures for Metrology

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#### ABSTRACT

A method for determining geometric process errors in MEMS devices from the resonant frequencies of simple structures is presented. The ability to accurately determine the as-built geometry of MEMS devices is important given the magnitude of the geometric uncertainty relative to the dimensions of these devices. As MEMS become smaller, the need for reliable metrology becomes even more important. The method presented here provides a way to determine the etch offset (the difference between the design width of a planar feature and the as-built width), the average angle of the side-walls and the compliance of the support of the structure. An important feature of the approach presented is that by using frequency ratios for various structural modes, neither the elastic modulus nor the mass density of the film need be known.

#### INTRODUCTION

The ability to measure the as-built geometry of micro-electro-mechanical systems (MEMS) is crucial in the analysis of their performance. Small variations from the designed geometry can arise due to mask misalignment, over or under etching, and other uncertainties in the fabrication process. An extensive list of examples chronicling the sensitivity of commercial and research devices to geometry variation has been compiled by Gupta [1]. In this same work, he addresses the problem of determining geometry variation. However, his method requires extensive finite element analysis and assumes *a priori* that the mechanical properties of the materials involved are well known. It has been well documented in the literature of MEMS material characterization that the mechanical properties of most thin films used are not known with precision. Indeed, for the Young's modulus of the most common MEMS material, poly-crystalline silicon, values ranging from 130 to 200 GPa have been reported within the last five years [2].

In this paper, we address the problem of determining variations from the designed geometry using lateral resonant structures made of materials whose mechanical properties need not be known. By comparing the resonant frequencies of structures whose nominal beam widths are different, the as-built beam width can be accurately determined. Unlike many *ex situ* conventional metrology measurements, such as SEM, surface profilometers, and interferometers, the *in situ* analytical metrology technique described here requires only the measurement of resonant frequencies of electrostatic comb drive resonators.

The basis for this approach is an analytic equation for the resonant frequency of a simple structure. Effects of support compliance are included and an examination of the sensitivity to uncertain measurements of frequency is presented.

#### OVERVIEW OF THE TECHNIQUE

The structures used in this approach are built of a single film of uniform thickness  $t$ , and consist of a large end mass of area  $A$  supported by two parallel cantilever beams of length  $L$  and

nominal width  $\hat{w}$ . Each structure, an example of which is shown in Fig. 1, is excited using an electrostatic comb drive [3].

The ideal results of an etch process for fabricating MEMS structures are perfectly straight and vertical side walls separated by the designed feature size. In reality, most etch processes suffer some degree of error from this ideal, both in terms of feature width and side walls that are not perfectly vertical. This leads to structures whose cross sections are not rectangular. Our work assumes that all structural cross sections are trapezoidal, with side wall angle  $\theta$ , and have an average width that differs from the design width by a “process offset”  $\varepsilon$ . The cross section of a typical beam is shown in Fig. 2.

The determination of the as-built geometry is based on the resonances of several different structures, each having the same thickness  $t$ , beam length  $L$ , and end area  $A$ , but each having a different nominal beam width  $\hat{w}_i$ . As shown in Fig. 2, the average width of the as-built beams will be  $w_i = \hat{w}_i + \varepsilon$ . Our subsequent analysis shows that the resonant frequency of such a structure has the form

$$f_i = \frac{\omega_i}{2\pi} = \sqrt{\frac{E}{\rho}} \varphi(t, \hat{w}_i, L, A, \varepsilon, \theta), \tag{1}$$

where  $E$  and  $\rho$  are the material’s Young’s modulus and mass density, and the function  $\varphi$  depends only on geometric quantities. The ratio of two resonant frequencies eliminates the material properties and leaves only geometry,

$$\frac{f_i}{f_j} = \frac{\varphi(t, \hat{w}_i, L, A, \varepsilon, \theta)}{\varphi(t, \hat{w}_j, L, A, \varepsilon, \theta)}. \tag{2}$$

Measurements of the resonant frequencies of at least three structures with different design widths allows Eq. (2) to be solved for the process errors  $\varepsilon$  and  $\theta$ .

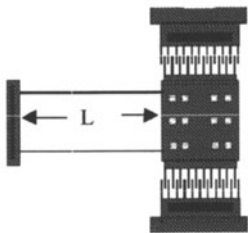


Figure 1. Dual beam structure.

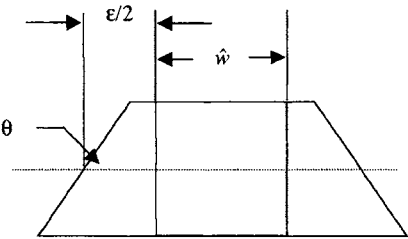


Figure 2. Trapezoidal cross-section of beam showing process errors