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Aluminum nano-cantilevers for high sensitivity mass sensors

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Abstract – We have fabricated Al nano-cantilevers using a very simple one mask contact UV lithography technique with lateral dimensions under 500nm and vertical dimensions of approximately 100nm. These devices are demonstrated as highly sensitive mass sensors by measuring their dynamic properties. Furthermore, it is shown that Al has a potential higher sensitivity than Si based dynamic sensors. Initial testing of these devices has been conducted using a novel scanning electron microscope setup where the devices were tested under high vacuum conditions. The Q-factor was measured to approximately 200 and the mass sensitivity was measured to 2 attograms/Hz by depositing electron beam induced carbon at the end of the nano-cantilever.

Index Terms – Nanoelectromechanical systems (NEMS), mass sensor.

I. INTRODUCTION

Cantilever based dynamic sensors have in recent years been a platform for highly sensitive mass sensors [1]-[3] and have demonstrated higher sensitivities than commercial Quartz microbalance technologies [4]. A cantilever based mass sensor works by measuring changes in its resonant frequency due to mass changes. Intuitively, by decreasing the size of the cantilever it is possible to measure smaller mass changes. The mass sensitivity can be derived using the equation for the resonant frequency and is given by [1]-[3],[5]:

$$\left| \frac{\partial m}{\partial f} \right| = \frac{2m}{f} \quad (1)$$

f is the resonant frequency and m is the mass of the resonator. This equation confirms that by decreasing the mass and increasing the frequency of the mechanical resonator the mass sensitivity is increased.

Current published works have reported attogram/Hz mass sensitivity [1],[3]. This has been possible by miniaturizing the cantilever by means of using the emerging nano-lithography techniques such as electron beam lithography on single crystal Si substrates [6],[7]. In this work we report on Al nano-cantilevers as a suitable replacement for Si based mechanical resonators. It will be shown that higher mass sensitivities can be achieved using Al and a very simple process flow is presented, which uses standard contact UV lithography to achieve sub 500nm

wide cantilevers with thickness of approximately 100nm. Finally, initial testing of the Al nano-cantilevers using a scanning electron microscope setup will be presented, which has been used to measure the devices resonant frequency, Q-factor and mass sensitivity.

II. DESIGN

By looking at the device as a whole, the cantilever must be excited into resonance in order to detect the change in resonant frequency. Making the cantilever very short would increase the mass resolution of a cantilever based sensor; however this is not a viable design. By decreasing the length of the cantilever the stiffness is increased and thus larger forces are needed for dynamic actuation. However, by rewriting the equation for the resonant frequency of a rectangular cantilever [7], it can be shown that the resonant frequency can be increased by decreasing both the Young's modulus (E) and the density (ρ) of the cantilever material, if the thickness (t), width (w) and spring constant (k) of the cantilever are unchanged.

$$f = 0.408 \frac{k^{2/3}}{t w^{2/3}} \cdot \frac{1}{\rho^{1/2} E^{1/6}} \quad (2)$$

TABLE I

The calculated length, resonant frequency and mass sensitivity for different cantilever materials. The cantilevers have a spring constant of 0.1N/m, a thickness of 100nm and a width of 1μm.

Material	E (GPa)	ρ (g/cm ³)	l (μm)	f (kHz)	$\partial m / \partial f$ (ag/Hz)
Al	74	2.70	5.70	2.61	1.18
Poly-Si	160	2.33	7.37	2.49	1.45
Si(100)	180	2.33	7.66	2.44	1.43
Ti	110	4.51	6.50	1.89	3.07
Cr	140	7.19	7.04	1.44	6.98
Ni	200	8.90	7.94	1.22	11.48
Au	80	19.3	5.85	0.96	23.19

In table 1 the mass sensitivity ($\partial m / \partial f$) is calculated for cantilevers with different materials. The spring constant of each cantilever is kept constant at 0.1N/m by adjusting only the length of the cantilever and maintaining a width of 1μm and a thickness of 100nm. By looking at table 1 it

is seen that Al has the best mass sensitivity followed by poly-Si and then single crystal Si. This is because Al has both a low density and low Young's modulus compared to the other cantilever materials. Among these materials, which are readily available for MEMS fabrication, Al is the best choice.

III. FABRICATION

In fig. 1 the fabrication of the Al nano-cantilevers is shown, which is based on a lift-off technique. The devices are realized on 4" Si wafers, fig. 1a. First, a resist mold is formed on the Si substrate using UV lithography, fig. 1b. Then, Al is deposited using e-beam evaporation, fig. 1c. Next, the Al deposited on the resist is removed by lift-off, fig. 1d. Finally, the metal structure is released by dry etching the underlying Si using an isotropic SF_6 based dry etch, fig. 1e.

Fig. 1. Process flow of Al nano-cantilevers. (a) silicon substrate. (b) photolithography is used to make a resist mold. (c) A 100nm thick Al layer is deposited. (d) the resist mold is etched, lifting off the Al on top of it. (e) the Si substrate is etched to release the Al structures.

The most critical step in the fabrication is the definition of the resist mold. In order to achieve sub 500nm cantilever widths a reverse lithographic process with AZ5214 photoresist was used [8]. Another important aspect of this process is that the release step is performed by dry etching, which alleviates stiction problems that are often seen using wet release techniques.

Fig. 2. SEM images of Al nano-cantilever before (a) and after (b) dry underetching.

In fig. 2 scanning electron images (SEM) are shown of the Al cantilever devices before and after dry underetching. From the images it is seen that the width of the nano-cantilever is below 500nm and the thickness is approximately 100nm. The length of the realized nano-cantilevers range from 1-10 μm .

IV. CHARACTERIZATION

In order to characterize the Al nano-cantilevers in a vacuum environment the devices were wire bonded to a PCB substrate and placed inside a SEM. Electrical feedthroughs in the SEM facilitate electrical connection to the Al nano-cantilever chips. On the chip an AC and DC voltage is applied between the driver electrode and the Al nano-cantilever (fig. 2b), which will actuate vertical motion. Vertical actuation is achieved because of two reasons; (1) the electric field is non-uniform above and below the cantilever due to the Si substrate and (2) the released nano-cantilever bends slightly upwards due to

internal stress, which also creates a non-uniform electric field. Two SEM based methods of measuring the frequency response of the nano-cantilever were used; (1) sweeping the actuation voltage frequency and imaging the nano-cantilever at a given actuation frequency and (2) focusing the e-beam on the cantilever and monitoring the secondary electron detector signal as a function of the actuation voltage frequency using a gain-phase analyser.

Fig. 3. SEM images of a nano-cantilever out of resonance (a) and in resonance (b). (c) the measured frequency response using the visual measurement method and a Lorentzian fit.

In fig. 3 two SEM images of an Al nano-cantilever are seen at two different actuation frequencies. In this experiment an AC actuation voltage of 9Vpp was used. In fig. 3a the nano-cantilever is not in resonance and the image of the cantilever is sharp, whereas in fig. 3b, at an actuation frequency of 1435kHz, the end of the cantilever is blurred due to the vibration. In fig. 3c the frequency response of this device is seen by measuring the amplitude directly from the images taken at different actuation frequencies. From the SEM images the vibrational

amplitude can be measured with an accuracy of approximately 30nm. In order to measure the Q-factor a Lorentzian fit was made and the Q-factor was extracted. In this case the Q-factor was measured to approximately 200. Considering the high vacuum conditions this Q-factor is very low. However, several factors can contribute to this, such as poor anchoring conditions due to underetching of the support (see fig. 2b) and surface losses due to the large surface to volume ratio of these nano-cantilevers [9,10].

By using the 2nd measurement method the frequency response was measured using the same device and is shown in fig. 4. In this measurement an AC voltage of 1V and a DC voltage of 10V was used. In fig. 4 it is seen that the detector signal shifts abruptly at the resonant frequency. The reason for this abrupt shift is not fully understood, however it could be due to a non-linear behavior that is seen for large vibrational amplitudes [11]. Another aspect of this measurement is that the resonant frequency is measured here to approximately double the first experiment. This is because in the first experiment the actuation of the cantilever was done with a pure AC signal between the driver electrode and cantilever, thus the AC frequency is half the electrostatic force frequency since the forces are proportional to the voltage squared. In the second experiment the DC voltage was much larger than the AC, thus the AC signal and the electrostatic force have the same frequency. The theoretical expected resonant frequency (neglecting spring softening effects) of an Al cantilever with these dimensions is approximately 3MHz, which confirms our measurements.

Fig. 4. Measured frequency response of the same device shown in fig. 3, using the electron detector signal versus the actuation frequency.

Fig. 5. SEM images of a nano-cantilever before (a) and after (b) carbon deposition and (c) the measured frequency responses both before (red) and after (black) carbon deposition.

V. MASS MEASUREMENT

A mass measurement has been performed by depositing carbon by electron beam induced deposition (EBID) [12]. In fig. 5 images of the Al nano-cantilever are seen both before (a) and after (b) the EBID. Also, the frequency responses of the nano-cantilever before and after are shown in fig. 4c. By measuring the frequency shift (6.5kHz) and using a rough estimate of the mass of the carbon deposit (14fg) the mass sensitivity of the device is calculated to approximately 2ag/Hz. The expected theoretical value, using equation 1, is approximately 1ag/Hz, which is in good agreement with the experiment. In this case the minimal frequency shift (df) that can be measured is approximately 500Hz, which leads to an absolute minimal detectable mass of approximately 1fg in these experiments. However, this number can be improved by improving the Q-factor and optimizing the electron detector signal with respect to noise.

VI. CONCLUSIONS

It has been demonstrated that using a relatively simple process, cantilevers with sub 500nm widths and 100nm thickness can be fabricated on a wafer scale using UV lithography. It was found that Al nano-cantilevers can be vertically excited into resonance with a lateral electrode by electrostatic actuation. Through a novel characterization setup, involving a SEM, the Q-factor was measured to approximately 200, which is lower than expected. However, more work needs to be done to investigate why, by improving current anchoring conditions. Finally, measurements of EBID carbon have been done and a mass sensitivity of 2ag/Hz has been measured. The SEM characterization technique has shown to be a very versatile and fast tool for measuring nano-cantilevers in vacuum condition. Furthermore, this work demonstrates that a very simple device can be used for high sensitivity mass measurements.

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