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Short Communication

A mechanical micro molecular mass sensor

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ABSTRACT

One of the bio-sensing mechanisms is mechanical. Rather than measuring shift in resonance frequency, we adopt to measure the change in spring constant due to adsorption, as one of the fundamental sensing mechanism. This study explains determination of spring constant of a surface functionalized micro machined micro cantilever, which resonates in a trapezoidal cavity-on Silicon <100> wafer, with the resonating frequency of 7000 cycles per second. This thin-flimsy-oxide micro-cantilever has a typical shape, and the tip of the micro-cantilever is dip-coated with chemically and biologically active material. The change in mass, due to adsorption, is detected by measuring the change in spring constant. The Force-Distance spectroscopy is used to detect the change in spring constant. The experimental results, show that the mechanical sensing scheme used, permit this surface functionalized micro machined micro cantilever to be used as a molecular mass sensor. The mechanical spring behaviour of a micro-cantilever, a micro-mechanical device can be used to develop ultra-tech micro-mechanical system using computer interface.

Keywords: *Micromachining; Micro-cantilever; Silicon<100>; Bio-Sensor; F-d Spectroscopy.*

INTRODUCTION

Designing Bio-MEMS/NEMS has always remained a critical task for the simple reason that the structures, on which the biologically or chemically active materials are dip-coated, smeared, spray-painted, and electro-deposited or sintered, should have bio-compatible surfaces. Normal choice of bio-compatible surfaces boils down to Silicon, Silicon Dioxide or Silicon Nitride. We have selected silicon Dioxide as a bio-compatible surface. This bio-compatible surface, upon surface treatments, becomes functionalized for linking to chemically or biologically active species, to use them as either sensors or detectors. This piece of work explains the design and fabrication of MEMS structure [1] [2] [3] – a simple micro cantilever, which is used for designing computer assisted Bio-MEMS applications.

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One of the bio-sensing mechanisms is mechanical. Rather than measuring shift in resonance frequency, we adopt to measure the change in spring constant due to adsorption, as one of the fundamental sensing mechanism. This study entails determination of spring constant of a surface functionalized micro machined micro cantilever, which resonates in a trapezoidal cavity-on Silicon <100> wafer [4][5][6], with the resonating frequency of 7000 cycles per second. This thin-flimsy-oxide micro-cantilever has a typical shape, and the tip of the micro-cantilever is dip-coated with chemically and biologically active material.

The change in mass, due to adsorption, is detected by measuring the change in spring constant. The Force-Distance spectroscopy is used to detect the change in spring constant.

EXPERIMENTAL

Micro-fabrication

Silicon<1 0 0 > n-type wafer is cleaned using Piranha cleaning procedure to remove contaminations. The Piranha cleaned wafer was then taken to a pyro-furnace for 700 nano-meters thermally grown oxide deposition, at $1100^0 C$, after calculating deposition parameters. The computed time was 1.10.11 Hrs using Modified Deal-Grove Time Calculations. The observed wafer deposition-colour was bluish-green. The wafer was then annealed for 1.5 Hrs to remove the stresses. After the wafer was drawn out of the oxidation furnace, the oxide thickness was measured to be 698 nanometres using Ellipsometer. The wafer was then given a Dehydration-bake at $120^0 C$ for 45 minutes on the heating iron. The MICROPOSIT® S-1813 positive photo-resist was dispensed at the centre of the wafer, using a dropper, on the wafer-on-the-chuck-of-spinner and spined at 500 rpm for 30 seconds and 3000 rpm for 2 minutes. After Positive Photo-Resist dispensing and spinning, the wafer was then PRE-BAKED at $70^0 C$ for 20 minutes and then taken for Photolithography using Karl-Suss® contact aligner for mask image transfer. The patterned wafer was then developed using a developer and observed under the microscope. The developed wafer was then micro-machined using 3:4 TMAH+WATER at 80 degrees centigrade. Wafer was intermittently observed under the microscope to ensure reliable machining. This micro-machined wafer was the tilted at 45^0 and Iso Propyl Alcohol was dispensed on the wafer slowly to ensure the release of the micro cantilevers. The chrome-gold sputtered layer improves adhesion of gold to the Silicon dioxide surface. Chrome-Gold was deposited using RF Magnetron sputtering. Substrate heating while chrome-gold sputtering, was not considered to be a necessary step. We have sputtered Chrome for 1 minutes and Gold for 3 minutes.

Surface Functionalization

The sputtered chrome-gold layer has affinity with thio-phenol molecules [7]. Considering this fact, we have dip-coated the piezo-resistive micro cantilevers with 1 micromole thio-phenol in ethanol solution for 3 Hrs. and then, rinsed with ethanol, for 2 minutes. The surface becomes functionalized for mass-sensing.

Reference Spring Cantilever Calibration With Atomic Force Microscopy

The spring constant of the unknown spring is calibrated by pressing it against a very stiff surface and then against a reference spring of known and lesser compliance [8]. The spring constant of the cantilever under test is then computed using the relation (1),

$$k_{unknown} = k_{std} \cdot [(InvOLS_{std} / InvOLS_{unknowm}) - 1] \quad (1)$$

In the above equation, $InvOLS_{unknown}$ is the inverse Optical lever Sensitivity (nm/Volt) for the cantilever under test measured on a very stiff surface and $InvOLS_{std}$ is the same quantity measured on a compliant surface with spring constant k_{std}. To determine the spring constant of the sample cantilever, the spring constant of the AFM cantilever must be known along-with the slope of the force curve. The slope of the force curve gives us the deflection sensitivity in nm/Volt. To compute the spring constant of the micro cantilever fabricated by us, we have used the relation (2),

$$k_{sample} = k_{AFM} [(DeflectionSensitivityofHard\,Region / DeflectionSensitivityofSoft\,Region) - 1] \quad (2)$$

Substituting the measured values of deflection sensitivities and known value of K_{AFM}, we have computed,

$$k_{sample} = 0.58 \cdot [(46.8/36.75) - 1] = 0.1622 Newton/meter$$

Computation of Spring Constant of Thio-Phenol treated Micro-Cantilever

With the above explained method, we have computed the spring constant of a surface functionalized micro cantilever, that reveal the change in the value of resonant frequency, in-turn, change in value of K,

For micro − cantilever1

$$k_{sample} = 0.58 \cdot [(46.8/46) - 1] = 0.0100 Newtons/meter$$

For micro − cantilever2

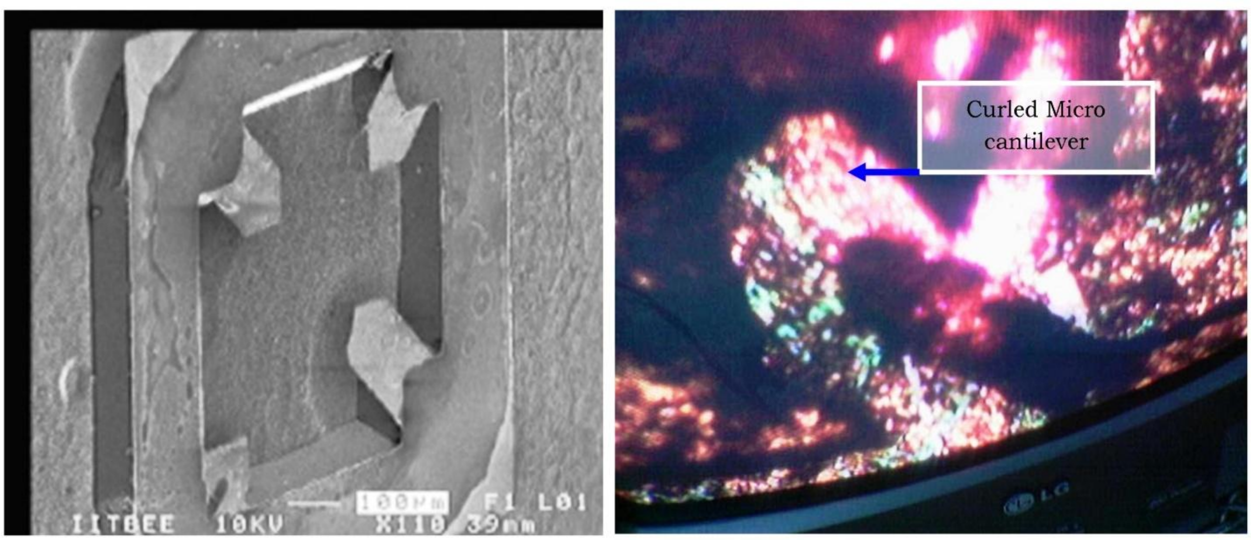
$$k_{sample} = 0.58 \cdot [(46.8/32) - 1] = 0.2682 Newtons/meter$$

For micro − cantilever3

$$k_{sample} = 0.58 \cdot [(46.8/41.5) - 1] = 0.0740 Newtons/meter$$

RESULTS AND DISCUSSION

Figure 1(a,b) depicts Scanning Electron Microscopy Micrograph and Atomic Force Microscopy Micrograph of the thio-phenol treated-gold functionalized surface of micro cantilever. Figure 2 depicts Atomic Force Microscopy Micrograph of thio-phenol treated gold surface scan showing clusters of thio-phenol molecule and Figure 3 indicates Surface topography of thio-phenol treated-gold surface. F-d spectroscopy of Micro cantilever before and after surface functionalization is depicted in Figure 4 and Figure 5. The investigation of F-d spectroscopy results reveals that, there is a change in the slope of the force-distance curve. Table 1 give a measure of change in slope is due to the change in spring constant and the adsorption of the thio-phenol molecule to the gold surface.



(a) Scanning Electron Microscopy Micrograph of Micro cantilever on silicon<1 0 0 > surface.

(b) Atomic Force Microscopy Micrograph of Surface Functionalized Micro-cantilever on silicon<1 0 0 > surface

Fig. 1. Surface Functionalized Micro cantilever.

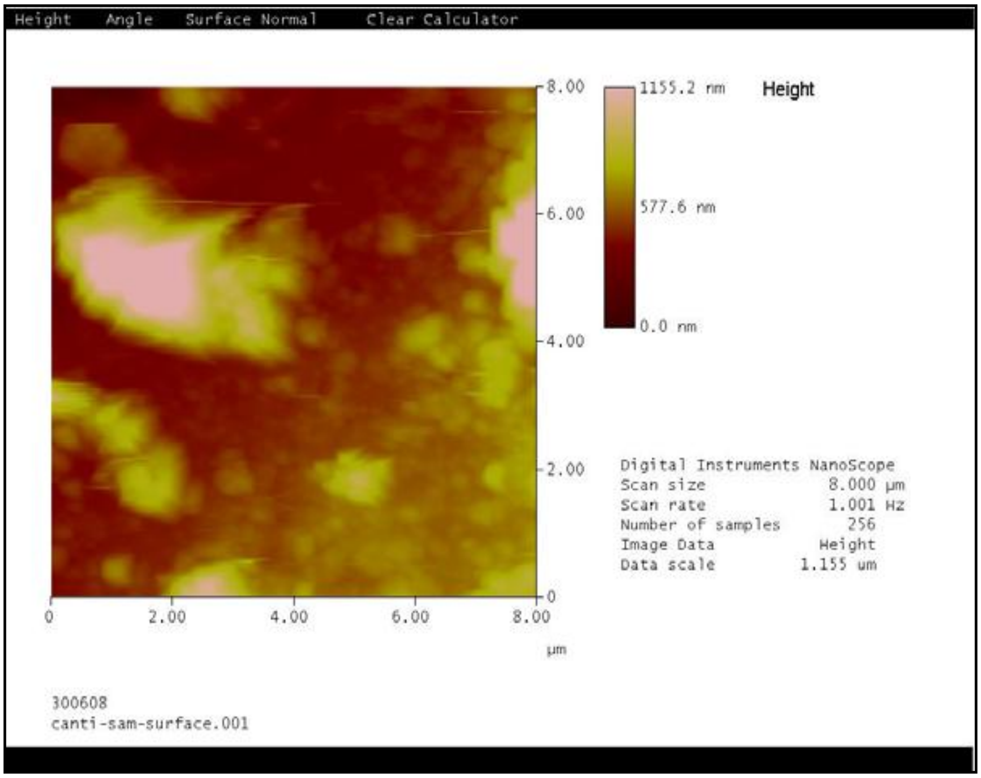


Fig. 2. Atomic force microscopy of thio-phenol treated gold surface on silicon <1 0 0 >. Scan showing clusters of adsorbed thio-phenol molecule

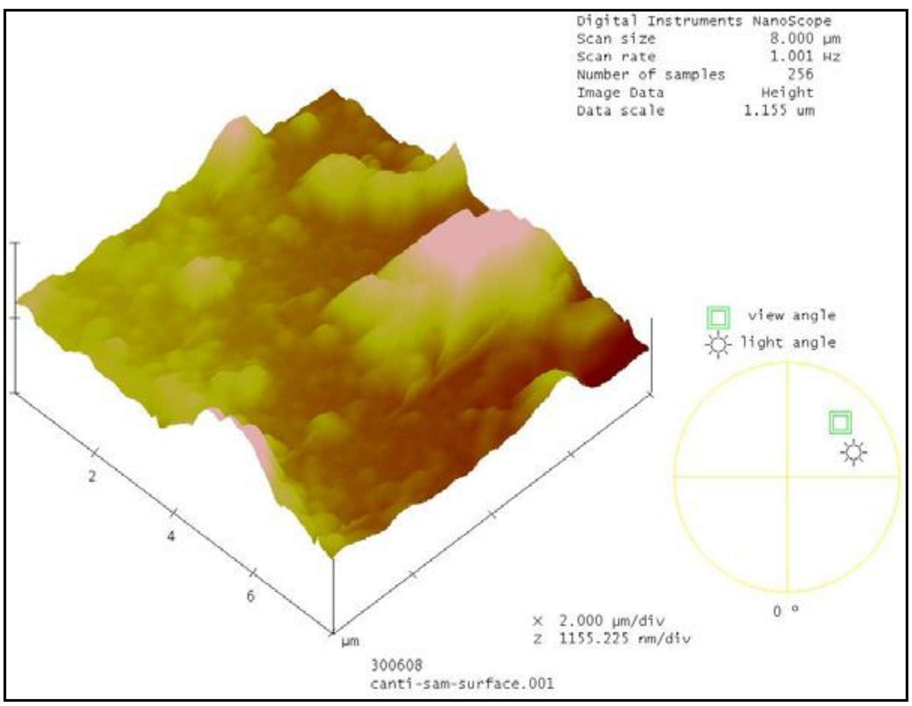


Fig. 3. Surface topography of thiophenol treated-gold surface

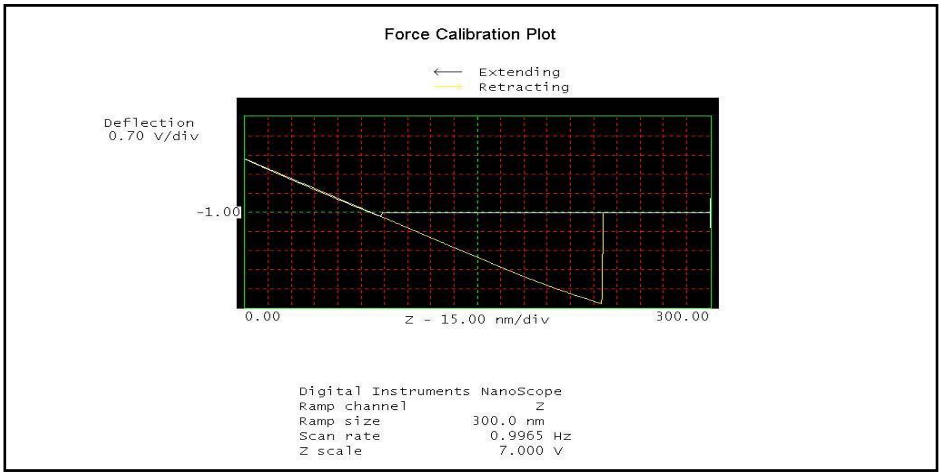


Fig. 4. F-d spectroscopy of micro cantilever before the surface functionalization.

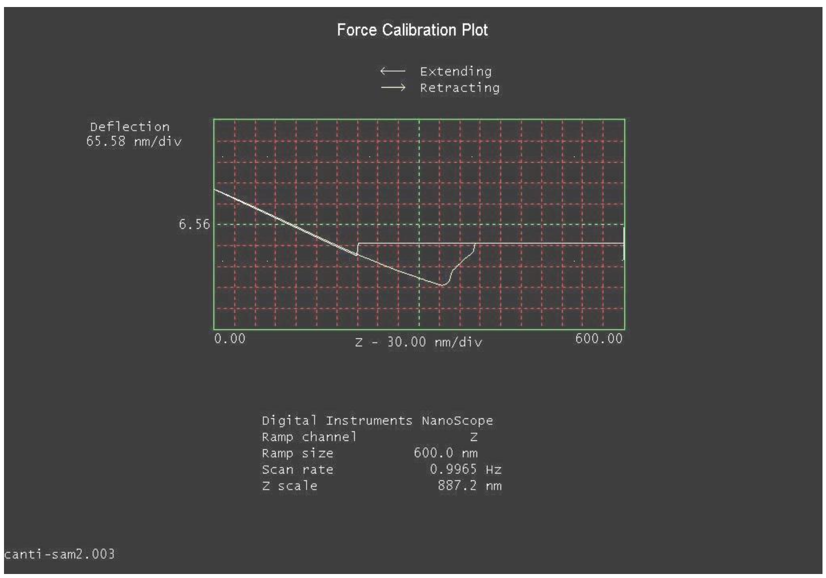


Fig. 5. F-d spectroscopy of micro cantilever after the surface functionalization.

Table 1. Spring Constant: Before and After Surface Functionalization

Sr. No.	Spring Constant of Micro machined Micro cantilever			
	Before Surface Function-alization	After Surface Function-alization	After Surface Function-alization	After Surface Function-alization
1	*0.16224 Newton/Meter*	*0.0100 Newton/Meter*	*0.26825 Newton/Meter*	*0.0740 Newton/Meter*

CONCLUSION

The change in mass, due to adsorption, is detected by measuring the change in spring constant. The Force-Distance spectroscopy is used to detect the change in spring constant. The study of F-d spectroscopy curves reveals that the surface functionalized micro cantilever behaves as a molecular mass sensor. The proposed device shall be used as micro-molecular mass sensor. The mechanical spring behaviour of a micro-cantilever, a micro-mechanical device can be used to develop ultra-tech micro-mechanical system using computer interface.

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