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Inverted tapered pillars for mass sensing

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ABSTRACT

Micro and nano resonators have been proposed as mass sensors with very high sensitivity. A special class of resonators, vertical micro-pillars, has been recently introduced with increasing mass detection capability, high sensor density and uniformity and reduced sensitivity to contamination and non-specific target absorption. Herein, we describe a newly conceived vertical micro-resonator with inverted tapered shape. With sensitivity of 33 Hz/fg and the reproducibility of 0.1 fg. The proposed geometry allowed the development of a new self-calibrating functionalization strategy which also makes our pillars very suitable for passing from liquid to dry environment. Selective adsorption of molecules is demonstrated and the application of the detector for a test target made of a self assembled monolayer of organic molecules is reported. The measured mass of the adsorbed molecules is 64 fg which corresponds to a density of 6.1×10^{14} mol/cm^2.

1. Introduction

The invention of atomic force microscope gave rise to the development of micro-cantilever technology which has soon revealed its potential for many different applications [1]. In the last few years outstanding results in sensing down to single molecule was reported [2]. Beside high sensitivity, other important features (e.g. small size, high parallelization, label free detection) make cantilever-like sensors an emerging tool in biology and diagnostics [3].

The basic principle behind cantilever sensing is that the adsorption of analyte changes the mechanical properties of the beam. In the dynamic mode, the loaded mass causes a change of the resonance frequency like in a quartz micro balance. However, due to the small cantilever mass, it is possible to achieve a sensitivity down to zepto gram (10–21 g) [4]. In many works the spring-mass model is used [1]. The resonance frequency is given

$$f_0 = \frac{1}{2\pi}\sqrt{\frac{k}{m^*}} \quad (1)$$

where k is the elastic constant, and m^* is the reduced mass of the cantilever. If a mass, Δm, is added the frequency changes according to Eq. (2):

$$f_0 = \frac{1}{2\pi}\sqrt{\frac{k}{m^* + \Delta m}} \quad (2)$$

Unfortunately, in the real world the adsorption do modify the mechanical properties of the micro-resonator in a more complex way. Firstly, the variation depends on the position where the adsorption takes place and the adsorbed molecules affect the elastic properties of the beam [5]. A more accurate equation is:

$$f_0 = \frac{1}{2\pi}\sqrt{\frac{k(\Delta m)}{m^* + \gamma\Delta m}} \quad (3)$$

where now k is a function of the adsorbed mass and γ is a geometrical parameter determined by the location of absorption. So it is clear that measuring only the resonance frequency does not provide quantitative any result. There are some approaches to address this issue, for example, to define lithographically a small adsorption spot (gold dot [2]) or, more commonly, to introduce a statistical analysis for adsorption events [6,7]. An alternative configuration consists in the fabrication of vertical microstructure (pillars) which oscillate around the normal to the wafer surface [8]. This simple variation it offers several advantages. The analyte can be localized right on the top of the pillar by a directional deposition system, such as thermal evaporation. The adsorbed analyte does not induce any stress on the oscillating part of the pillar. As a consequence, the Eq. (2) can be correctly applied in order to associate the change in frequency with adsorption. Furthermore, the fabrication process is intrinsically symmetrical, so that all the vertical walls are equally finished and no asymmetrical residual stresses are induced by fabrication as in the case of horizontal geometry. The uniformity of the pillars geometry and mechanical properties depends only on controllable processes such as electron beam lithography and reactive ion etching. There are no stiction effects when used in liquid/dry

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environment. Finally, as it will be described below, the deposition of a known amount of material on the top of the pillar can be used for absolute calibration of the sensor. This also permits to introduce a self-calibrating functionalization strategy.

2. Fabrication

The fabrication scheme consists in two main processes: definition of the cross section by lithography and deep anisotropic etching. In addition, there are also some cleaning and surface preparation steps.

The starting material is a (1 0 0) oriented, single side polished, P-type silicon wafer. After piranha (H_2O_2(35%):H_2SO_4 = 1:3 at 90 °C) and HF cleaning a 100 nm silicon dioxide layer is grown by PECVD in order to protect during the fabrication process the portion of the substrate which will be the top area of the pillar. The sample is spin coated with 500 nm of poly-methylmethacrate (PMMA) 950 K resist (4000 rpm) and baked for 10 min at 180 °C.

The pillar in-plane rectangular geometry is defined by e-beam lithography (Zeiss Leo 30 keV) with typical size is 5×3 µm. We chose rectangular cross section because the spectrum of mechanical response shows well defined and separated peaks [1]. After PMMA developing, a 20 nm nickel layer is evaporated by means of e-beam and the Ni mask for the subsequent dry etching is obtained through a lift off process by removing the resist in hot acetone.

Before the etching, oxygen plasma is performed in order to remove the residual resist and argon plasma is used to define better the metal mask. We have developed a Bosch™-like process to obtain a deep etching for both silicon and silicon oxide with an Inductively Coupled Plasma reactor (ICP, STS-Surface Technology). For passivation, we use a plasma of mixture of C_4F_8 and Ar (100 and 20 sccm) at a pressure of 7 mTorr and with 600 W of RF power applied to the coil. For etching we use a plasma of mixture of SF_6 and Ar (110 and 20 sccm) at a pressure of 8 mTorr and with 600 W of RF power applied to the coil and 50 W to the platen. Many cycles are executed to remove silicon to create the vertical resonator. The duration of the process settles the height of the pillar. Typically, we remove almost 15 µm which correspond to 48 cycles. Differently from Ref. [8], we want avoid a strictly vertical profile. So the etching process has been optimized to obtain a slight, well controlled undercut (≈3°, see Fig. 1 inset). This geometry shows several advantages: the sensor mass is reduced (about 36%) without changing the sensitive area; the evaporation on the top is better defined because of the intrinsic shadowing effect; the oscillation amplitude is increased, due to the thin pillar base; the stress induced by the oscillation is confined on the pillar base, which is less affected by the thermal drift induced by the laser which is used for monitoring the motion of the pillar.

The final step it is the removal of the metal mask and protective silicon oxide providing a clean and flat silicon surface for the next functionalization process. First, the metal mask is removed by a 15 min dipping in piranha solution, then the oxide is dissolved in hydrofluoric acid. Since the spontaneous re-oxidation of silicon is a very slow reaction which lasts for weeks, the mass of the pillar continuously increases thus inducing a constant drift of the resonance frequency. In order to avoid this effect, we speed up the oxidation reaction to obtain a stable oxide coating. For this aim, we have explored different ways: O_2 plasma, thermal oxidation or chemical passivation with piranha solution. We observed that, for our purpose, all methods are essentially equivalent so we choose the last one for practical reason.

This fabrication procedure permits to obtain inverted tapered pillars (Fig 1). We fabricated on our chip 12–24 sensing elements, but in principle, due to small surface area of pillars, it is possible to design devices with a much larger number of sensors.

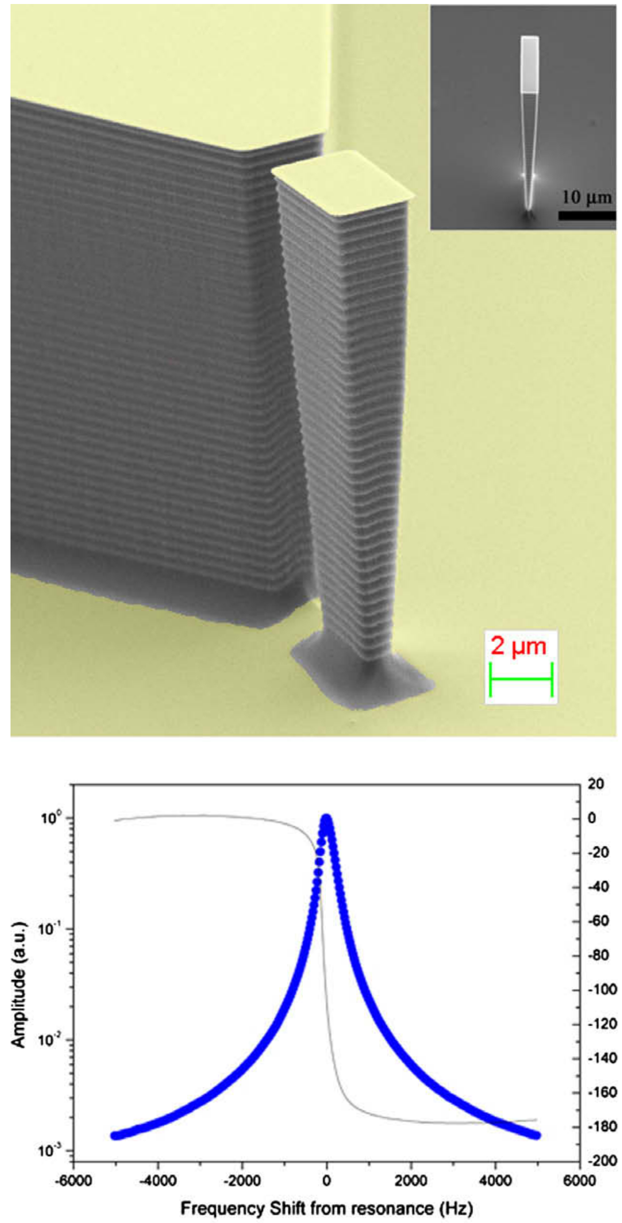

Fig. 1. Scanning electron microscope image of a 15 µm-height pillar after gold deposition. The gold layer is colorized to show that lateral wall and the base are not coated. On the bottom there is a typical resonance curve. The resonance frequency is 5,010,750 Hz. Inset: side view of the pillar which better illustrates the undercut effect (≈3°). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

3. Experimental setup

We have built an experimental setup to measure the resonance frequency of micro-pillars in vacuum by using the optical deflection method [9].

The device is placed into small vacuum chamber, evacuated with a turbo – rotary pump system with base pressure better than 1×10^{-6} mbar. The pressure is constantly monitored with a full scale gauge, because measurements can be compared only if are taken in the same condition due to the dependence on pressure of the mechanical properties of a cantilever. A laser (DPSS @ 532 nm) is focused to a spot of few microns by using a long working distance microscope objective with 0.4 numerical aperture. The