CO-RESONANT CANTILEVERS AS HIGHLY SENSITIVE MASS SENSORS

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ABSTRACT

Cantilever-based mass sensors offer the potential of high sensitivity but also require very sophisticated configurations to reach the femtogram range and below. In this contribution, the potential of a co-resonant principle for dynamic mode cantilever sensors is evaluated for that purpose. The approach relies on mechanical coupling and eigenfrequency matching of a micro- and a nanocantilever. As a consequence, the coupled state allows for reliable oscillation detection using common laser deflection, while also providing high sensitivity to mass loads. The sensor fabrication process as well as initial experimental studies of the mass sensing potential are presented.

KEYWORDS

Cantilever sensors, co-resonance, mass sensitivity, batch fabrication, silicon nitride

INTRODUCTION

The introduction of a co-resonant state is a promising approach for enhancing the sensitivity of dynamic-mode cantilever sensors. This concept is based on mechanical coupling and eigenfrequency matching of a micro- and a nanocantilever, which enables the combination of both resonators' respective advantages: The microcantilever's potential for robust and reliable oscillation detection by established laser-based methods used in commercially available measurement equipment (e.g. AFM), and the nanocantilever's high sensitivity for external interactions (force, mass load) due to its low stiffness and effective mass resulting from nanoscale dimensions [1,2].

Co-resonance is achieved when the eigenfrequencies of both cantilevers differ by approximately less than 20 % and the resulting sensor properties are defined by a complex interplay between the specific matching state and the individual resonators' properties [3]. Hence, for a sensing application, the cantilevers must be designed in such a way that a desired sensing performance can be realized. This is accomplished by selecting dimensions that allow for (i) high sensitivity (mainly influenced by the nanocantilever's properties), and (ii) the predefined eigenfrequency matching state. Particularly the latter requires a fabrication process that enables precise control of the dimensions of both cantilevers.

These design considerations are all based on analytical calculations. Previous work has therefore primarily focused on investigating the underlying theoretical principles of co-resonant cantilevers for sensing applications, specifically the relations between different sensor properties, such as sensitivity, linearity, and noise [3,4]. These fundamental studies provide the basis for practical sensor use cases. The first advancement reported here is the implementation as a mass sensor.

Cantilever-based mass sensing is primarily utilized in biological applications, such as monitoring of cell growth [5-8], and in gas sensing, e.g. the detection of specific molecules such as volatile organic compounds [9]. Typically, silicon-based materials and carbon nanotubes (CNTs) are employed for cantilevers. Their dimensions range from a few to several hundred micrometers in length, and thicknesses are below the single-digit micrometer range to provide high sensitivity through low stiffness (typically 1 mN/m to 10 N/m). Consequently, the resonance frequencies are in the order of several kHz to a few MHz.

Oscillation detection is most commonly achieved through laser deflection, and in fewer cases by interferometry. Very high sensitivity has been reported for a CNT cantilever in combination with a radio receiver for oscillation detection. This highly sophisticated approach was tested with gold atoms and a sensitivity of (0.1 – 1) zeptogram has been achieved [10]. For silicon microcantilevers, attogram sensitivity has been reported for detection of virus particles in air and with laser-doppler vibrometry [8]. These are two studies with highly specialized and tailored setups that are not compatible with standard mass-sensing applications. For the latter purpose, reported mass sensitivities typically lie in the order of picogram [5,7,11].

The co-resonant principle offers a comparatively simple approach that is compatible with standard measurement equipment relying on piezo-actuation and laser-deflection readout. Here, we are reporting initial experimental studies where a sensitivity in the femtogram range with a microcantilever stiffness of few N/m has been achieved.

EXPERIMENTAL

Sensor fabrication

The studied sensors have been fabricated from silicon nitride with the newly developed process depicted in figure 1 [12]. It is based on standard microfabrication techniques and overcomes the challenges associated with co-resonant sensor structures by enabling (i) the realization of predefined eigenfrequency matching states and sensor properties, (ii) very high aspect ratios of the nanocantilever (lengths of several ten micrometers and thicknesses below 100 nm) beneficial for the sensor's sensitivity, and (iii) very good reproducibility and accuracy of sensor properties throughout the wafer [12]. With this process, co-resonantly coupled sensors with desired properties can be batch-fabricated for the first time.

The co-resonant structures in the mass sensing studies feature a rectangular cross section of both resonators and are fabricated with dimensions for a target value of 0 % eigenfrequency deviation. The basis is a 380 µm thick silicon wafer with 1 µm low-stress silicon nitride (SiN) on front side and back side. This initial SiN layer defines the thickness of the microcantilever. To achieve low stiffness and consequently high sensitivity, the nanocantilever target thickness is set to 100 nm. The other dimensions of both cantilevers are calculated to result in a theoretical matching state of 0 % eigenfrequency deviation. Furthermore, the design is guided by the following considerations:

- Minimum microcantilever length and width of 100 µm and 30 µm, respectively, to enable reliable oscillation detection by laser deflection.
- Aspect ratio of nanocantilever that can be realized without bending (based on studies reported in [12]).

These boundary conditions lead to the dimensions of an exemplary sensor listed in table 1. Due to variations within the base wafer and process parameters, the resulting sensors show various degrees of deviation from the target values, as also indicated in table 1. However, this is still well within the range for co-resonant behavior.

Vibration experiments

The mass sensing potential of the fabricated sensors is investigated by vibration experiments conducted in a scanning electron microscope (SEM) using a home-built piezo stage (figure 2 and details in [1]). It comprises an SEM sample stub with a copper

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base plate and shear piezo with an aluminum sample holder on top. As depicted in figure 2a, the sensor is placed on the sample holder in such a way that the movement of the shear piezo plate translates into a sensor vibration in its preferred oscillation direction. In this configuration, the electron beam is providing a side view of the sensor structure where the oscillation envelope and amplitude are clearly visible as shown in figures 2c and d.

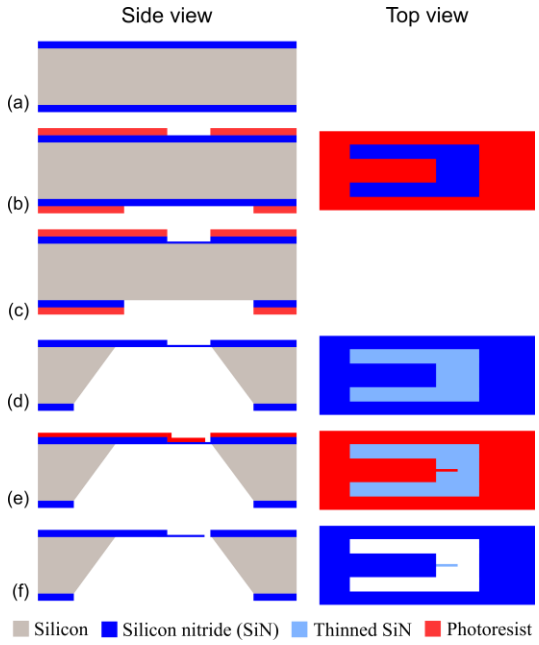

Figure 1: Fabrication process flow for co-resonantly coupled sensor in silicon nitride: (a) Initial silicon wafer with both sides coated with 1 µm low-stress silicon nitride. (b) Application of photoresist on both sides of the wafer and photolithographic definition of microcantilever outline and nanocantilever area (front side) and complete sensor area (back side). (c) Thinning of nanocantilever area to desired thickness (front side) and complete etching of silicon nitride to expose silicon (back side) with reactive ion etching. (d) Stripping of photoresist and KOH etching of back side silicon to suspend the silicon nitride membrane. (e) Front side lithography for definition nanocantilever length and width. (f) Structuring of nanocantilever and release of the complete sensor structure by reactive ion etching and subsequent photoresist removal by oxygen microwave plasma ashing (previously published under CC BY license in J Micromech Microeng [12]).

This setup enables continuous and real-time observation of the sensor's oscillation behavior and the determination of amplitude response curves. The latter allows the derivation of deposited mass by considering the shift of the coupled system's resonance frequencies. Therefore, the piezo is excited with a sinusoidal signal from a signal generator and amplitude response curves for both resonance peaks of the coupled system (f_a an f_b) are obtained by sweeping the excitation frequency around the predetermined resonance frequencies and capturing a video of the cantilever oscillation. Automated frame-wise post-processing enables deduction of the corresponding amplitude response curves exemplarily depicted in figure 3.

During each frequency sweep, amorphous carbon material is deposited in all locations irradiated by the electron beam due to remaining gas molecules and contaminants inside the SEM chamber. This additional mass is mainly influencing the highly sensitive nanocantilever and altering its eigenfrequency, resulting in a change of the coupled system's resonance frequencies due to the co-resonant state. Hence, the additional mass on the nanocantilever can be quantified from the resonance frequency shifts and based on the coupled system's properties (geometry and spring constants) determined from SEM images right after sensor fabrication and the initial vibration measurement.

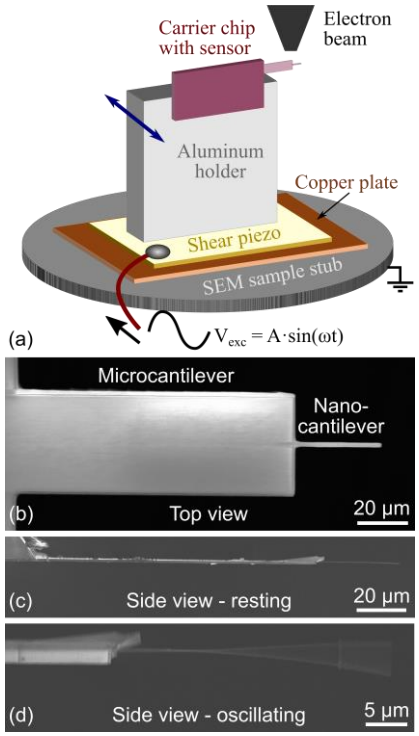

Figure 2: (a) Sketch of vibration setup for sensor characterization. The blue arrow indicates the oscillation direction excited of the shear piezo. (b) - (d): Scanning electron microscope images of a co-resonant sensor made from silicon nitride: (b) top view, (c) and (d) side view at rest and excited at one of the coupled resonance frequencies (f_{exc} = 110.82 kHz; V_{piezo} = 20 mV), respectively. In (d) only the free end of the microcantilever and the nanocantilever are shown for clarity.

*Table 1: Properties of exemplary co-resonant mass sensor. (*Calculated from dimensions as they cannot be measured due to the coupled system being fabricated as a whole. **The coupled system's resonance frequencies have been determined from the SEM experiments; initial values before any mass deposition.)*

Property	**Microcantilever**	**Nanocantilever**
Length	112 µm	35.2 µm
Width	39.4 µm	2.6 µm
Thickness	1 µm	0.103 µm
Spring constant	1.62 N/m	3.9 mN/m
Eigenfrequency*	123.63 kHz	127.98 kHz
Matching state	3.5 %	
Coupled state	***f*a (left peak)**	***f*b (right peak)**
Res. freq.**	102.567 kHz	110.931 kHz

RESULTS AND DISCUSSION

Figure 3 depicts amplitude response curves obtained from SEM images of the exemplary sensor shown in figure 2 (properties in

table 1). During the experiment, the excitation frequency was always swept with increasing direction first, and followed by a decreasing sweep. Please note that only the curves for the increasing sweeps are depicted in figure 3 for clarity. Furthermore, first the left resonance peak (lower frequency, f_a) was studied by six consecutive sweeps followed by the same number of frequency sweeps for the right resonance peak (higher frequency, f_b). The resulting resonance frequency obtained from the SEM images as well as the calculated mass loads are summarized in table 2.

The calculation of the added mass is based on the general relation between resonance frequency f, spring constant k and effective mass m_{eff}:

$$f = \frac{1}{2\pi}\sqrt{\frac{k}{m_{eff}}} \qquad (1)$$

However, for the co-resonantly coupled system, *effective* values for spring constant and effective mass need to be considered. This is due to an inherent feature of the co-resonance: a single cantilever can be described as a harmonic oscillator. When two cantilevers are coupled and frequency matched, the resulting coupled harmonic oscillator model exhibits two resonance peaks. Each of these can in turn be represented by a single harmonic oscillator but with effective properties [3]. These are dependent on the eigenfrequency deviation of both cantilevers, their individual spring constants and effective masses and can be calculated from the basic geometric dimensions, initial vibration experiments and known material properties of the used low-stress silicon nitride [3,13]. Furthermore, we have made the following assumptions to enable the calculation of the mass load:

- The effect of the deposited material on the nanocantilever's spring constant is negligible.
- The deposited material on the microcantilever is much smaller than its mass and does neither affect its stiffness nor its effective mass.

With these considerations, the deposited masses can be calculated based on equation (1) and results in the values listed in table 2. Please note that the listed values reflect the total accumulated mass and not the changes for each frequency sweep.

Table 2: Measured resonance frequencies for both peaks and calculated total amount of deposited mass (amorphous carbon) after each frequency sweep. Please note that the mass calculation assumes that the added material does not influence the spring constant of the nanocantilever.

*f*a (left peak)	*f*b (right peak)	Mass in fg
102.57 kHz	---	0
102.37 kHz	---	140.7
102.24 kHz	---	233.6
101.63 kHz	---	686.7
101.42 kHz	---	842.9
100.85 kHz	110.83 kHz	1273.4
---	110.81 kHz	5465.3
---	110.71 kHz	29425.9
---	110.67 kHz	38352.4
---	110.59 kHz	57425.1
---	110.56 kHz	65471.8

From this data, the mass sensitivity S of each resonance peak can be determined by plotting the resonance frequency shift versus the accumulated mass and applying a linear regression fit. For the exemplary sensor shown in figure 2 and table 1 it amounts to S_a = 1.35 Hz/fg and S_b = 0.004 Hz/fg for left (f_a) and right (f_b) resonance peak, respectively.

The difference between the sensitivities of the two resonance peaks is a consequence of the co-resonant coupling. As stated above, the effective properties of each peak in the coupled system are defined by the individual cantilever's features, but with different contributions. Thus, while one resonance peak is more dominated by the properties of the microcantilever, the second one is more influenced by the nanocantilever [3].

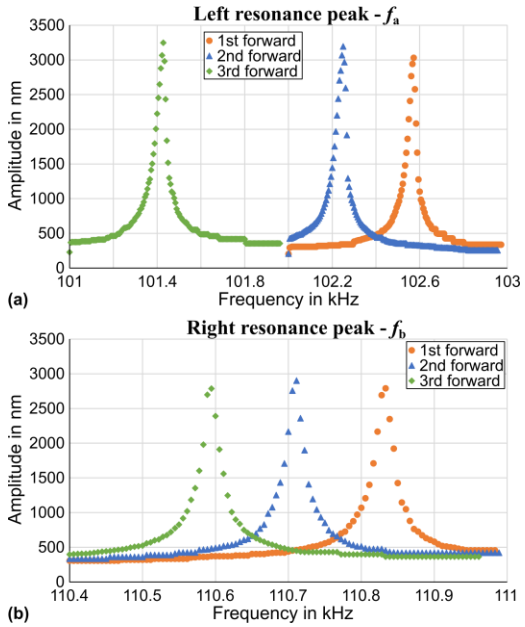

Figure 3: Amplitude response curves for both resonance peaks (fa, fb) of the coupled sensor from figure 1 obtained from SEM images for several sweeps with mass deposition (only selected curves are shown for clarity).

CONCLUSION AND OUTLOOK

The presented work employs the co-resonant principle for cantilever-based mass sensing. It is demonstrated that femtogram sensitivity can be achieved with a silicon nitride-based microcantilever with a stiffness of few N/m fabricated by standard microstructuring techniques.

These initial studies clearly indicate the tremendous potential of the co-resonant concept to enable highest mass sensitivity without the need for sophisticated oscillation detection and with sensors that can be batch-fabricated with application-specific properties by standard techniques. Future research will focus on investigating the mass sensing capabilities of co-resonant sensors with different frequency matching states and comparison to the theoretical model predictions as well as exploring the potential limit of detection for these sensors.

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