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Columnar shaped microresonators for mass detection and gas analysis

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ABSTRACT

In this paper miniaturized silicon columns mounted perpendicular to a surface are presented as microresonators for the detection of an exceedingly small mass. In a first sensor design the column resides on a rigid silicon substrate and is forced to oscillate in the first flexural eigenmode. Mass sensitivity was investigated loading a single latex sphere on the top surface of the column and detecting the frequency shift of the resonant frequency. Oscillation was monitored using the optical beam deflection method, focusing a laser beam at the same surface. However, optical readout was substantially hampered by scattering of the laser beam at the loaded particles and diffraction at the column surface. In a second optimized design a silicon column was mounted on a flexible silicon nitride membrane. In this way the optical readout was applied from the flip side of the membrane completely separating the load area and the optical beam path. A proof of principal is given for the optimized sensor design investigating the influence of pure gases on the extrinsic damping behavior of the membrane columnar oscillator.

Micro- and nanomechanical resonators have been introduced as tools for the detection of mass, vapor, gas viscosity, fluids, and chemical reactions with significant sensitivity [1–8]. In particular mass detection is an attractive application in life science, offering the capability of a selective and label-free detection of biological species, e.g. single cells or viruses, by chemical tailoring the surface properties [9–12]. The optimization of mass detection sensitivity of resonant sensors requires to maximize the eigenfrequency ω_0 and quality factor $Q = \omega_0/\Delta\omega$ with the linewidth $\Delta\omega$, and at the same time to reduce their effective resonator mass m_{eff} by miniaturization [13]:

$$\Delta m = 2m_{eff}\sqrt{\frac{B}{Q\omega_0}}10^{-DR/20}.$$

B and DR denote the measurement bandwidth and the dynamic range of the electronic equipment, respectively. Various sensor geometries have been proposed, e.g. conventional cantilevers, u-shaped cantilevers, double clamped bridges, microshells, etc. [14–16]. Almost all of them were realized applying micromachining processes known from nano- and microelectromechanical system manufacturing. The demand of miniaturization requires intricate lithography and etching processes to realize systems with dimensions below some micrometers. In this paper, we present columnar shaped microresonators on substrates and thin membranes. This particular geometry guarantees a simple manufacturing route with the capability to integrate the resonator structure into a lab-on-a-chip environment. Columnar probes were applied for the detection of a concentrated mass as well as for measuring the molecular mass of pure gases.

One of the advantages compared to the fabrication of the intricate resonator structures mentioned above is the simple fabrication scheme of columnar sensors [17]. Fabrication is based on 385 μm thick, (001) oriented monocrystalline silicon wafers. In the first process wafers were spin-coated with a 1.4 μm thin photo-resist layer (AZ5214E). An elongated ring like structure was transferred into the photo-resist by conventional UV lithography and subsequent development with AZ400K. It defined the lateral geometry of both the trench as well as the column resonator in the center part and served as a hard mask layer. Manufacturing was accomplished by strong anisotropic reactive dry etching in an inductively coupled plasma (ICP) etcher, applying the so called Bosch process. Due to the repeated etching and passivation steps during this process, the side walls of the columnar resonator features corrugations in the order of 200 nm. The height of columns is determined by the etch time. With this fabrication sequence it is possible to control both the lateral dimensions as well as the column height on a micrometer scale, using conventional UV photolithography and subsequent ICP etching. Finally, the photo-resist layer was stripped yielding silicon columnar microresonators in a trench, protected by the substrate from mechanical damage. A cross sectional scheme and a scanning electron

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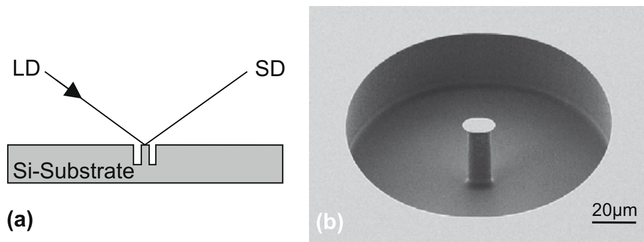

Fig. 1. (a) Cross sectional scheme of the silicon column as microresonator on the rigid silicon substrate. Optical readout is applied from the column side (BD beam deflection laser, SD split diode). (b) SEM image of a columnar microresonator with a cross section of 9 × 14 μm² and 40 μm height.

microscopy (SEM) image of a silicon column on a rigid substrate are shown in Fig. 1. The stadion like cross sectional geometry of the column was chosen to force the vibration direction of the first flexural eigenmode perpendicular to the long axis. This facilitates the alignment of the optical detection scheme presented in the following. Fig. 2a shows a finite element simulation (Comsol Multiphysics) of the first flexural eigenmode of the column assuming that the substrate is rigid.

All measurements were performed applying the optical beam deflection method, that was introduced by Meyer et al. for atomic force microscopy applications [18]. For this purpose the beam from a laser diode (LD: power <1 mW at $\lambda = 635$ nm) is expanded and then focused onto the sample surface, to obtain a focus diameter in the order of 10 μm (see Fig. 3). The laser beam is reflected from the top side of the column under an angle of 45° and detected with a split photo-diode (SD) (SSO-DP-3.22-6, Silicon Sensor GmbH, Germany). The difference signal of the diodes is amplified and fed to a network analyzer (Agilent 4395A). For mechanical excitation the sample chip is glued to a shear piezo (P) that is driven by the network analyzer (NA) via a homemade low impedance amplifier. Spectra are recorded on a PC and evaluated, fitting a Lorentzian peak to the resonance spectra.

With the presented columnar dimensions the resonant frequency of the first flexural mode is typically in the order of 4–6 MHz with a quality factor Q of 1500–2500 in air. First mass loading experiments were carried out loading a latex sphere of (2.5 ± 0.1) μm diameter (Micromod GmbH, Germany) on the top of a column with lateral dimensions of 10 × 20 μm² and 42 μm height. Its resonant frequency of 4.48 MHz was determined keeping the sensor in air at ambient conditions. For the loading of a latex sphere, a glass fiber was thermally pulled down to a tip diameter of ca. 10 μm. A droplet of the latex sphere dispersion was placed onto a cover slip. After evaporation of the dispersant, the very end of the fiber was used to pick up a number of latex spheres. The loaded fiber was clamped to a three axis micromanipulator, to carefully pull off a single sphere at the top edge of the column. These experiments were performed under control of a homemade magnifying optics. From the evaluated resonance spectra, before and after loading, a −11 kHz frequency shift was observed (Fig. 4). A sensitivity of approximately (0.8 ± 0.1) Hz/fg was deduced from the loaded mass ((8.4 ± 1.0) pg). The typical noise level observed in the experiment results in a mass detection limit of 25 fg for the given column geometry [16].

In this columnar resonator design the top surface is not only used for loading mass, but at the same time for the optical reflection of the laser beam (see Fig. 1a). However, scattering at the loaded mass and diffraction at the top column surface results in the diminution of the detected signal.

An optimized sensor was established, separating the optical detection path from the loading area of the column. It consists of a clamped silicon nitride membrane carrying the silicon column on its center (Fig. 5a). Beam deflection is now performed on the flip side, reflecting the focused laser beam at the bottom of the column, whereas mass loading still takes place on the top of the column. To

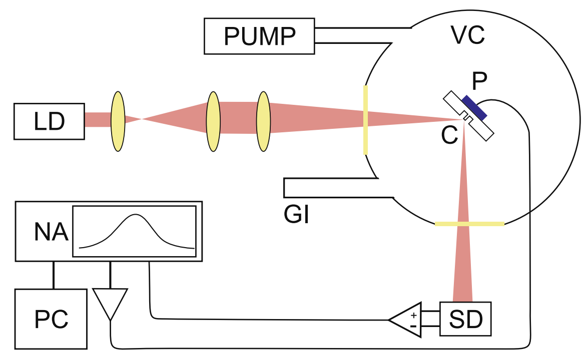

Fig. 3. Experimental set-up for the phase sensitive measurement of the columnar microresonator (C) vibration. The vacuum chamber (VC) is primarily used for the investigation of the influence of pure viscous gases (GI: gas inlet) on the damping behavior of the resonator.

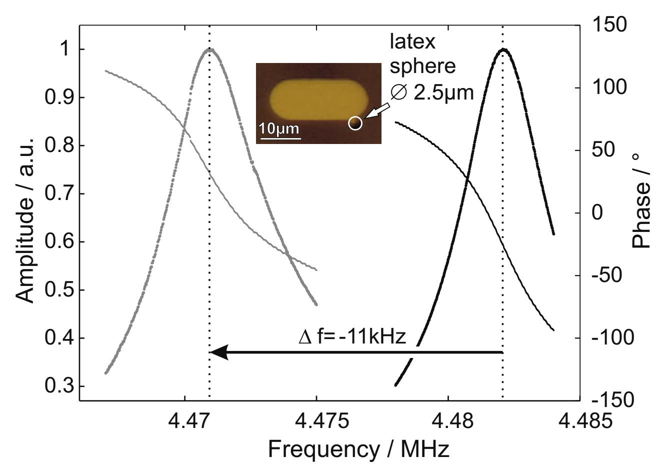

Fig. 4. Measured resonance spectra of the first flexural mode before and after loading a latex sphere. The inset shows an optical transmission image from above with the latex sphere attached to the top edge of the columnar microresonator.

Fig. 2. Finite element simulation of the first flexural eigenmode of a silicon column on (a) a rigid silicon substrate and (b) a 2 μm thick silicon nitride membrane clamped at the circumference. The dimensions are in accordance with the geometrical data of the sensors from the SEM image in Fig. 1and optical image in Fig. 5, respectively.