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High-frequency micromechanical columnar resonators

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Abstract
High-frequency silicon columnar microresonators are fabricated using a simple but effective technological scheme. An optimized fabrication scheme was invented to obtain mechanically protected microcolumns with lateral dimensions controlled on a scale of at least 1 μm. In this paper, we investigate the influence of the environmental conditions on the mechanical resonator properties. At ambient conditions, we observed a frequency stability $\delta f/f$ of less than 10^{-6} during 5 h of operation at almost constant temperature. However, varying the temperature shifts the frequency by approximately -173 Hz °C^{-1}. In accordance with a viscous damping model of the ambient gas, we perceived that the quality factor of the first flexural mode decreased with the inverse of the square root of pressure. However, in the low-pressure regime, a linear dependence was observed. We also investigated the influence of the type of the immersing gas on the resonant frequency.

Keywords: microresonator, microcolumn, frequency stability, mass sensor, resonators in viscous fluids, molecular mass, cantilever

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Cantilever probes and several types of micromechanical resonators like bridges and microcolumns are commonly used as sensors for various applications, e.g. ultrasensitive mass detection, measurement of viscosity, or identification of biological species [1–5]. In mass detection applications, the eigenfrequency of a chosen eigenmode decreases with increasing effective resonator mass. Upon loading a mass m_c on top of a circular columnar resonator of mass m_0, length l, and radius r, the resonant frequency f_1 of the first flexural mode shifts to [6]:

$$f_1' = \frac{f_1}{\sqrt{1+\frac{m_c}{0.24 m_0}}} \quad \text{with} \quad f_1 = \frac{1}{2\pi}\sqrt{\frac{3\pi E}{4\cdot 0.24\rho}}\cdot\frac{r}{l^2}, \qquad (1)$$

where E is Young's modulus and ρ the mechanical density of the resonator material. In order to improve mass resolution, it is reasonable to reduce the mass of the resonator by miniaturization. Some reported approaches used sophisticated fabrication techniques and sensor geometries [7–9]. We have introduced a simple and reliable process to produce silicon columnar resonators oriented perpendicular to a plane substrate [10]. With these columnar resonators we demonstrated mass detection at a sub-picogram level with a typical sensitivity of 1 Hz fg^{-1} in air.

In this paper, we introduce an improved sensor geometry by shielding the fragile column from mechanical damage. This is accomplished by removing a ring shaped structure from the substrate forming a cylindrical column in the center of a groove. In order to avoid an influence of the shielding wall on the resonator properties, the width w of the groove was chosen to exceed the penetration length μ of the acoustic wave [11]:

$$\mu = \sqrt{\frac{\eta}{\pi\rho f_1}} \qquad (2)$$

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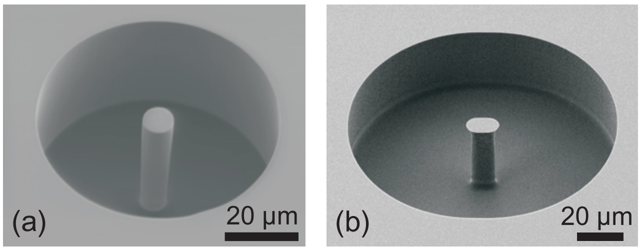

Figure 1. Scanning electron microscopy images of ICP-etched silicon columnar resonators of 40 μm height. Two exemplary cross sections of columns are presented: (a) circular (diameter 8 μm, groove width 29 μm) and (b) oval (short axis dimension 9 μm, groove width 50 μm).

emitted from the vibrating column at ambient pressure. In case of air as immersing gas ($\rho = 1.204\,\mathrm{kg\,m^{-3}}$, $\eta = 17.1\,\mu\mathrm{Pa\,s}$) the penetration length μ is approximately 1 μm for a typical resonator of 5 MHz resonant frequency, whereas groove width was chosen between 18 and 50 μm.

All applications mentioned above require the measurement of an exiguous shift of the resonant frequency. However, the immersing gas, as a viscous fluid, damps the vibration. The resulting shift and broadening of the resonance peak reduce the quality factor Q and reduce the measurement accuracy of the resonant frequency. This dissipative process depends mainly on the fluidic properties of the immersing gas. Therefore, we studied, in this paper, the influence of environmental parameters, such as pressure, temperature and type of the immersing gas.

2. Technological scheme

Columnar sensors were fabricated from (001) oriented silicon wafers. After Radio Corporation of America (RCA) cleaning, the wafers were spin coated with an about 1.5 μm thin photo resist layer (AZ1518). Ring-like structures were defined by UV lithography and the exposed photoresist was removed with the developer AZ 400 K. The center part of each ring defined the cross section of the columns and worked as hard mask in the subsequent anisotropic inductively coupled plasma (ICP) etching process. The etching time was chosen to obtain columns of nominal 42 μm height. However, due to the ICP reactive ion etching lag, the etching depth was 40–42 μm for groove widths 18–50 μm. Figure 1 shows scanning electron microscopy (SEM) images of two typical columnar sensors. Due to the oblique imaging in the SEM exacted by the sensor geometry, the height of columns could only approximately be determined with an accuracy of about $\pm 1\,\mu$m.

3. Mechanical characterization

For the mechanical characterization, a shear piezo actuator was glued with conductive silver to the bottom of the silicon chip carrying the columns. The piezo actuator is driven by a network analyzer (HP 4195A) via a home-made high-frequency low-impedance amplifier. The mechanical vibration of the column is detected applying the widespread optical beam deflection method known from atomic force microscopy (AFM). The beam of a laser diode (wavelength 635 nm, power 1 mW) was focused under an angle of about 45° on top of the column to a spot size of approximately 5 μm, and the reflected beam was detected using a split photodiode. This differential photodiode signal, obtained due to the periodic tilting of the column during vibration, was fed to the network analyzer after amplification with a home-made transimpedance amplifier. The quality factor $Q = f_1/\Delta f$ was deduced from measured spectra defining the frequency difference at the 3 dB amplitude drop on each side of the resonance peak as the width Δf of the resonance curve.

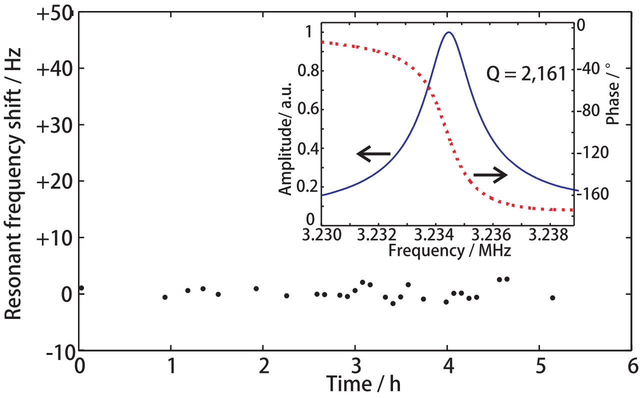

Figure 2. Temporal fluctuation of the resonant frequency f_1 of a circular column resonator (diameter 8 μm, height 40 μm) at ambient conditions (temperature 22.6 ± 0.5 °C and normal pressure). The frequency deviation from the average resonant frequency $\bar{f}_1 = 3\,234\,369$ Hz is plotted on the ordinate. The inset displays typical spectra of the amplitude and phase.

Irrespective of the application, the frequency fluctuation from the mean resonant frequency $\bar{f}_1$ limits the smallest detectable frequency shift. Therefore, the resonant spectra of a circular column (diameter 8 μm, height 40 μm) were measured at constant temperature 22.6 ± 0.5 °C and ambient pressure during 5 h of operation. A typical spectrum of the circular resonator is shown in the inset of figure 2 revealing a quality factor of 2161 with an average resonant frequency of $\bar{f}_1 = 3\,234\,369$ Hz. From the evaluated resonant frequency of 27 spectra (see figure 2) a standard deviation of $\delta f_1 = 1.2$ Hz was deduced. This resulted in a frequency stability $\delta f_1/\bar{f}_1$ of less than 10^{-6}. Additional measurements (not shown) for three days of operation without external stabilization indicated a long-term frequency stability of $\leqslant 10^{-4}$. Thus even with uncontrolled laboratory conditions, mass detection in the range of a few hundred femtogram is feasible.

The pressure dependence of the resonant frequency and the quality factor of common cantilever sensors have been described in the literature [12–14]. However, it is not obvious whether the same theory can also be applied to the columnar sensors, because they do not obey the assumption of the cantilever model (length $\gg$ width $\gg$ thickness). According to the description of the cantilever behavior by Zhang *et al* [15], the pressure range can be separated in four different regimes corresponding to the dominant damping mechanism: (i) fluid damping in the viscous regime, (ii) rarefied gas damping in the transition regime, (iii) molecular damping in the molecular

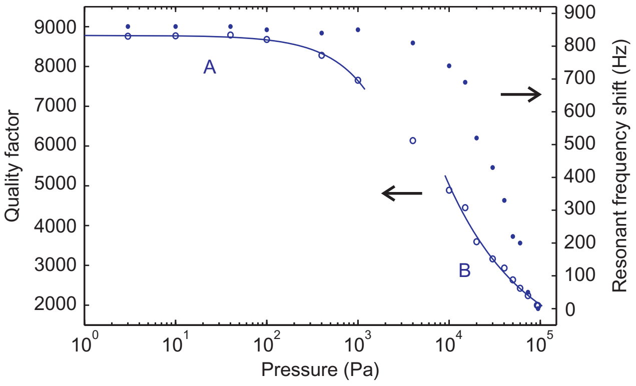

Figure 3. Pressure dependence of the quality factor (open circles) with least square fits (line) for the two distinct pressure regimes. Fit equation for (A) $Q = 8787 - 1.9485 p/Pa^{-1}$ ($R^2 = 0.994$) and (B) $Q = 440432 \cdot \frac{1}{\sqrt{p/\mathrm{Pa}}} + 618$ ($R^2 = 0.987$). The resonant frequency shift (filled circles) is depicted with respect to the resonant frequency $f_1 = 4\,447\,960$ Hz at ambient conditions.

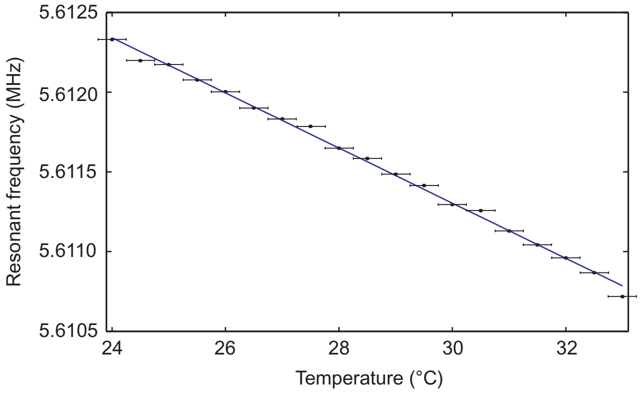

Figure 4. Temperature dependence of the resonant frequency of an oval resonator (smaller axis dimension 12 μm, height 42 μm) in N_2 (5.0 purity) at ambient pressure. The confidence interval $\pm 3\sigma$ of the frequency measurement was ± 3.4 Hz.

regime, and (iv) intrinsic damping in the high vacuum regime. Most commonly the Knudsen number, defined as the ratio of the molecular free mean path and the characteristic dimension of the resonator, is used to identify the different regimes. However, the boundaries between the regimes are vague and cannot be determined precisely. According to Judge *et al* [14], the viscous damping model works accurately for a Knudsen number smaller than 0.001 and in some cases even up to 1.

For a circular microcolumn (diameter 8 μm, height 40 μm, $f_0 = 4\,447\,960$ Hz at ambient conditions) the pressure dependence of both the resonant frequency and quality factor in air was investigated as can be seen from figure 3. By fitting the quality factor, we could distinguish two regions in the covered pressure range. For a pressure in the range 10–100 kPa the Knudsen number was estimated to be smaller than 0.1. In this region, the quality factor Q is proportional to the inverse of the square root of the pressure p that corresponds to the established viscous damping model [14]. However, in the low-pressure range (up to 1 kPa) the measured quality factor shows an almost linear behavior $Q \sim Q_{\mathrm{int}} - a \cdot p$ with positive constants Q_{int} and a. That means the quality factor Q of the fluid and resonator as a coupled system approaches the intrinsic quality factor Q_{int} of the resonator if the pressure is reduced by a certain amount. We would like to point out that in this low-pressure range, the mean free path of gas molecules in the groove exceeds the groove width and might lead to ballistic energy transport. This will be investigated in future studies.

The experiments with columns of circular cross section revealed that the direction of vibration was uncorrelated to the shear force direction of the piezo actuator. This behavior hampered the alignment of the optical detection set-up. Therefore, an oval cross-sectional geometry of the resonator was introduced with the nominal axis ratio of 1 : 1.5 (figure 1(b)). For these columns, the degeneracy of the first flexural mode of the circular columns is lifted, and the fundamental flexural eigenmode has a direction of motion perpendicular to the long axis. ICP etching of these oval resonators gave an undesired underetching of the hard mask pad caused by an imperfect thermal contact of the wafer with the electrode of the reactor (figure 1(b)). Regardless of this deviation from the intended geometry, the mechanical properties of the silicon resonators were unaffected. Therefore, we pursued the thermal characterization of microresonators.

Statistical temperature fluctuation is a main source of an unwanted frequency shift. Its influence on the resonant frequency was investigated varying the chip temperature by 10 °C in steps of 0.5 °C. Measurements were performed in a vacuum chamber, immersing the column in nitrogen at ambient pressure to reduce the influence of moisture. Evaluation of the measured spectra revealed an almost linear drop of the resonant frequency with temperature with a gradient of $-173\,\mathrm{Hz}\,{}^{\circ}\mathrm{C}^{-1}$ (figure 4). This is in good agreement with the behavior of conventional silicon cantilevers investigated under ultrahigh vacuum conditions, for small temperature variations around room temperature [16].

An additional experiment was conducted to identify the influence of the focused laser beam of the beam deflection system on unwanted heating of the silicon column. Reducing the laser power from 1 mW to 100 μW revealed a distinct shift of the resonant frequency of 48 Hz. However, with the typical response time of our complete detection scheme of approximately 3 ms we were not capable to resolve the time evolution of this frequency shift. This means that laser heating takes place on a time scale below 3 ms as an upper limit of the thermomechanical time constant of the columnar microresonators.

Damping of a resonator depends not only on the external thermodynamic state variables, but also on the type of the immersing gas. The viscosity η of gases is weakly affected by the type of gas [17]. However, the gas density ρ is proportional to its molecular mass M. To study this effect in more detail, we immersed an oval-shaped column in various pure gases (molecular mass added in brackets): krypton (83.798 u), argon (39.948 u), oxygen (31.999 u), nitrogen (28.013 u), methane (16.043 u) and helium (4.003 u). Frequency spectra were recorded at ambient conditions (figure 5(a)). In accordance with the findings of Xu *et al* [17] for viscous damping of cantilevers, we confirmed that the evaluated resonant

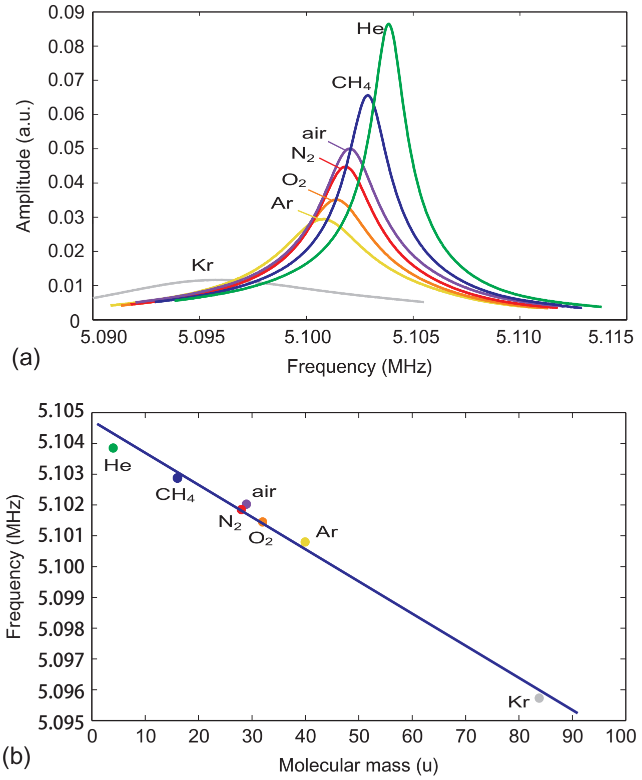

Figure 5. a) Measured resonant spectra for an oval columnar microresonator (smaller axis dimension 12 μm, height 42 μm) immersed in different gases. The precision of the frequency and amplitude is ±3.4 Hz and better than 10 %, respectively. (b) Measured resonant frequency f_1 as function of the molecular mass (in atomic units).

frequency of our columnar microresonators also dropped linearly with increasing molecular mass of the immersing gas (figure 5(b)). We could also verify this dependency in air, assuming its averaged molecular mass as 28.959 u.

The slope of the linear fit curve of 104 Hz u^{-1} exceeds that found by Xu *et al* by almost a factor of 20. This, together with the confidence interval of the frequency measurement of $\pm 3\sigma = \pm 3.4$ Hz, theoretically leads to the smallest detectable change in molecular mass of $\Delta M = 5 \times 10^{-3}$. The fit quality, expressed in terms of the correlation coefficient R^2 of 0.987, indicates that in practice, there are uncertainties presumably with respect to the purity of gases, the leakage rate of our home-made vacuum chamber, and a small error in the pressure measurement ($\Delta p = \pm 100$ Pa@10^5 Pa). Furthermore, it is not obvious that the linearity claimed in the theory of viscous fluids holds without taking into account the chemical behavior of the gas species expressed in terms of interaction cross sections. However, the salient sensitivity makes the columnar resonators an interesting sensing tool for various applications like the measurement of the dynamic viscosity η/ρ of gases, temperature and pressure.

4. Conclusions

We studied the mechanical behavior of columnar microresonators with a resonant frequency of the first flexural mode in the range of 3–6 MHz. Room-temperature measurements revealed a relative frequency variation of this eigenmode less than 10^{-6} for 5 h of operation, which gives the confidence interval $\pm 3\sigma$ of frequency measurements of approximately ±3.4 Hz. Varying the pressure of the ambient gas revealed two different regimes. For the pressure range of 10–100 kPa, the quality factor of circular resonators was proportional to the inverse of the square root of pressure, which is an indication of viscous damping in the immersing gas. With decreasing pressure, a linear dependence of the quality factor was observed that converges against the intrinsic quality factor of silicon resonators in vacuum. The resonant frequency dropped linearly with increasing temperature at ambient conditions, in accordance with the thermomechanical properties of silicon. Furthermore, operating the microresonators in different pure gases at ambient conditions showed a linear dependence of the quality factor with the molecular mass of the immersing gas, as expected from the model of a viscous medium.

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