Resonance frequency behavior of silicon nitride cantilevers as a function of pressure in different gas environments

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Abstract—**We present experimental data on resonant behavior of the first flexural mode of a silicon nitride cantilever in noble gas ambients of He, Ar, and Xe. To this aim thermal noise spectra have been measured with an optical setup. Overall resonance frequency and the quality factor of the first flexural mode vs. pressure are in good agreement with the existing theories for the molecular and viscous pressure regimes. Prior to the viscous regime we observe a small anomalous increase in the resonance frequency which is most pronounced in the He environment. The increase points to a slight stiffening of the cantilever. Surprisingly temperature increase from laser irradiation shows a further increase in the stiffening. Diffusion of gas in the near-surface region of the amorphous cantilever material may lead to stiffening and so account for the small frequency increase.**

I. Introduction

Nano-electromechanical systems (NEMS), in particular cantilever devices, are suitable candidates for ultra sensitive mass detection [1], [2], detection of individual components in a gas mixture [3]–[6] and absolute pressure measurements [7]. The main motivations of cantilever application as selective mass sensors are the small size,the ultra-high sensitivity and the integration of transducer and readout on-chip [8]–[10]. With proven detection sensitivity down to sub-attogram it is important to understand the underlying physics of resonant behavior and explore the potential in different application fields. Side effects as impact of temperature and pressure [11], adsorption-induced variation in the stiffness of a cantilever [12] and collisional interactions of the surrounding gas molecules could affect the dynamical behavior of cantilevers while shrinking their size. Therefore these side effects should be understood properly for future applications of cantilever devices.

Here, we present an extension of our previous work on the effect of pressure on the Q-factor and the resonance frequency of silicon nitride (SiN_x) cantilevers in air [13]. We showed that the resonant behavior is in accordance with the existing models for molecular and viscous pressure regimes. However a small anomalous increase of the resonance frequency was observed in the transition from the molecular to the viscous regime. In the present work we explore the physical background of this anomalous increase in more detail. To exclude beforehand any surface-chemical interaction noble gas ambients are used in the present work. The anomaly was observed by others in laterally vibrating silicon cantilevers coated with 100 nm gold [3].

There the frequency is ascribed to a temperature effect due to the current flow in piezoresistive detection. Important evidence for the temperature argument was the absence of this increase for uncoated silicon cantilevers when using optical detection where heating is possibly less important [14], [15].

II. Fabrication and measurement

Cantilevers are fabricated from home-made Low-Pressure Chemical Vapour Deposited (LPCVD) SiN_x [16] on silicon $< 100 >$. The cantilevers are 200 nm thick, 100 μm long and 17 μm wide. Device fabrication includes e-beam lithography and dry etching as described elsewhere [17] . Fig. 1a shows a typical cantilever.
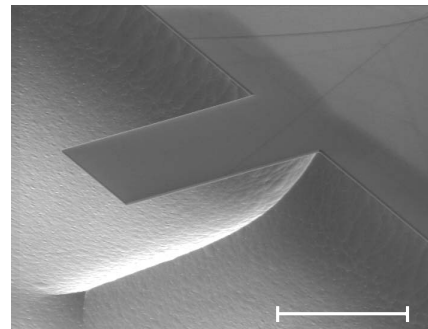

Fig. 1.   SEM picture of a SiN_x cantilever. Scale bar corresponds to 20 μm.

The resonance frequency is measured in a home-made optical laser deflection setup extended with an appropriately windowed vacuum chamber pumped by a combination of a dry fore pump and a turbo molecular pump [17]. The chamber is pumped down to a pressure of 1×10^{-6} mbar after which the first measurements are carried out up to 1×10^{-5} mbar, while pumping the chamber continuously. Then the pump is turned off while the pressure stays constant for further measurements at higher pressures. The pressure readout is done by a combination of a Pfeiffer full-range pressure meter for the range from 1×10^{-6} to 1×10^{-4} mbar and 2 Varian absolute pressure meters (baratron); one for the range from 1×10^{-4} to 1×10^{-2} mbar and one for the range from 1×10^{-2} to 1000 mbar. The resonance spectrum of a cantilever is monitored by a spectrum analyzer (Agilent 4395A) with a smallest resolution

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bandwidth of 1 Hz. The resonance frequency and the Q factor are determined from Lorentzian fits (red line) through the data, as illustrated in Fig. 2.
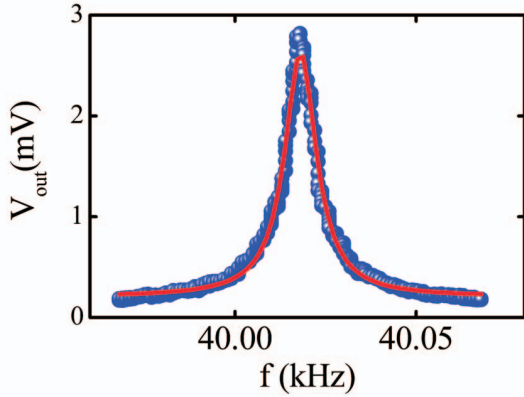

Fig. 2. Thermal noise data of the first bending mode of a cantilever. The red line is a Lorentzian fit through the data defining the position of the resonance and the corresponding Q-factor.

III. Q-FACTOR AND RESONANCE FREQUENCY VS PRESSURE

Q-factor and resonance frequency behavior vs pressure in He, Ar and Xe are shown in Fig. 3 and 4 respectively. The frequency axis is normalized to the highest frequency corresponding to the lowest pressure. For comparison the results from the previous work on cantilevers in air are included. On the upper horizontal axis the corresponding Knudsen (Kn) number is shown. This number is the ratio between the mean free path of the gas molecules and the beam width w. Three different regimes can be distinguished, as indicated by the vertical dashed lines. At very low pressures (Kn >10) the Q-factor is dominated by intrinsic losses (intrinsic regime). With increasing pressure the molecular regime starts at about Kn ≈ 10 [18]. Momentum exchange between individual gas molecules and the cantilever at a rate proportional to the difference in velocity between the molecules and the resonator is dominant damping source in this molecular regime. When Kn < 0.01 [18] gas molecules can no longer be considered as free molecules and collisions between the molecules have to be taken into account. In this regime the gas behaves like a viscous incompressible fluid (viscous regime). The solid lines in Fig. 3 and 4 represent the result of calculations using explicitly the different molar masses and viscosities [19].

For all gases the Q-factor behavior meets the analytical models sketched above (Fig. 3). As for the frequency behavior in air we notice a close correspondence between experimental and calculated data (Fig. 4). A small deviation is observed for the noble gas ambients. The experimental decrease of frequency vs pressure is systematically faster than the theoretically predicted. It happens typically in the pressure range between the molecular and viscous regime where no analytical description is available for.
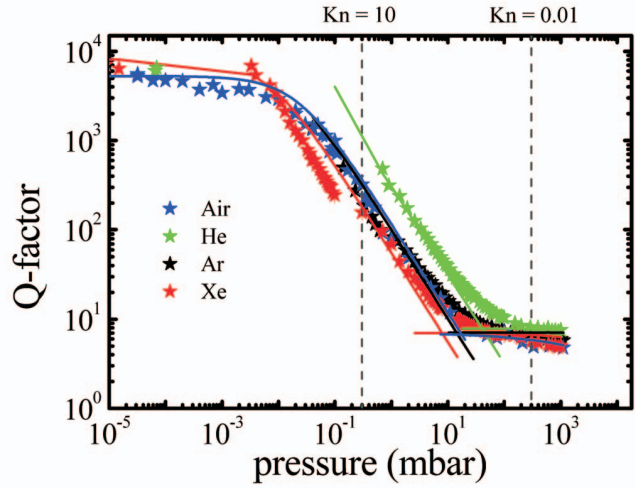

Fig. 3. Measured data of Q-factor vs pressure for four different gas environments. The Solid lines correspond to the theoretical models showing the good agreement with the experiment.
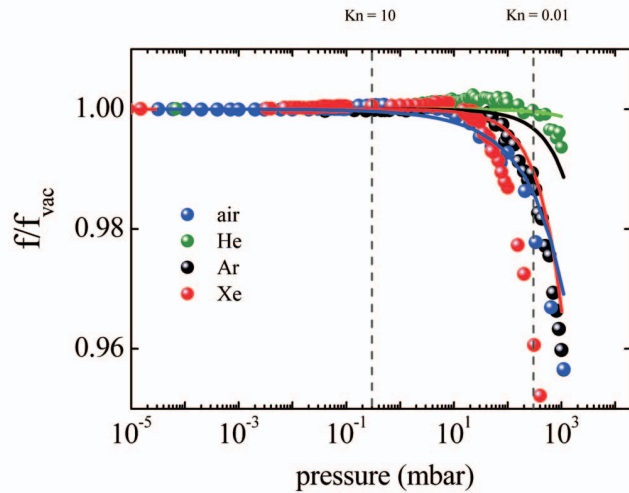

Fig. 4. Measured data of the relative resonance frequency shift vs pressure for four different gases. The Solid lines correspond to the theoretical models showing close agreement with the experiments.

When zooming in around the molecular flow regime an anomalous increase in the resonance frequency shows up prior to the steep decrease discussed above, which is not predicted by the theory so far (see Fig. 5). Apparently the He peak shows the highest increase in the relative resonance shift. The other noble gases (Xe, Ar) show almost the same frequency increase compared to air but with the maximum at different pressures. If temperature would play a role here one would expect indeed He to show the largest effect because of its superior cooling ability. To verify the possible impact of temperature measurements at 2 different laser powers have been performed. The result is shown in Fig. 6. At higher laser

power the anomalous increase of resonance frequency is even larger. Using testoterm clock indicators (detection capability $\geq 40°C$) it has been verified that at a laser power of 1.6 mW the temperature increases from < 40 °C (atmospheric) to 43-46 °C (10^{-5} mbar). For laser power of 3.1 mW the temperature increases from 65-71 °C (atmospheric) to 88-93 °C (10^{-5} mbar). With higher temperatures a lower stiffness is expected [11], [14], [20], but surprisingly an increase (and so a stiffening) is observed instead. This observation rules out the role of temperature to explain the anomalous frequency increase. Given that the largest increase in frequency is observed for the smallest gas atom (He) a different explanation may applies based upon gas diffusion into the near-surface region. It is envisioned that filling up cavities in the amorphous compound may give a slight stiffening. Penetration is expected to be most significant for the smallest gas atom (He). However it is unclear what the role of temperature is in this respect. On one hand a temperature increase will gives enhanced diffusion (stiffening), but on the other hand also a stronger out-gassing (softening). Apparently the results from different laser power point to a predominant diffusion effect.

Fig. 6. Resonance frequency vs. pressure for two different laser powers in Xe environment. ●: 1.6 mW, ●: 3.1 mW.

IV. CONCLUSION

The experimental behavior of the Q-factor and the relative resonance frequency shift vs. pressure in He, Ar and Xe are in qualitative agreement with the analytical models for molecular and viscous regime. An unexpected slight increase in the resonance frequency is observed between the molecular and the viscous regime and can not be accounted for by the impact of temperature. An alternative explanation based upon a stiffening due to gas diffusion into the near-surface amorphous cantilever material is proposed.
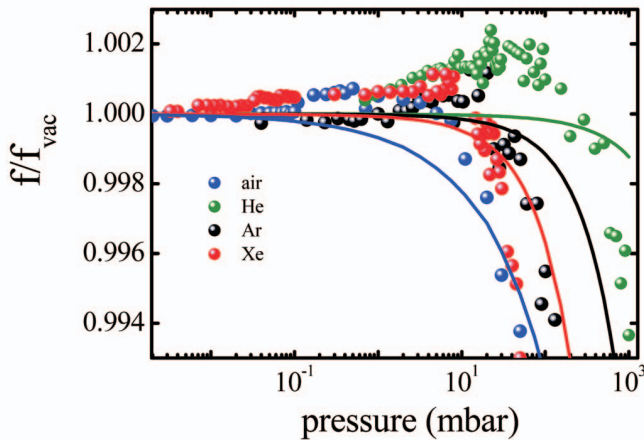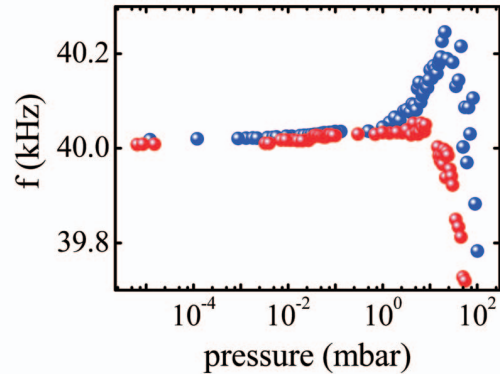
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Fig. 5. Measured data of Q-factor and the relative resonance frequency shift vs pressure in He, air, Ar and Xe environment (dots). The solid lines represent analytical models.

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