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Position-dependent mass responsivity of silicon MEMS cantilevers excited in the fundamental, two-dimensional roof tile-shaped mode

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Abstract
The potential of a special class of eigenmodes of microcantilevers, the so-called roof tile-shaped modes, is investigated for resonant mass sensing. Due to the 2D shape of vibration, the mass responsivity (resonance frequency shift per unit mass change) of these modes depends on the position along the length and width of the cantilever. This is in contrast to the commonly used 1D eigenmodes, where the mass responsivity is only dependent on one spatial variable. We mapped the position-dependent mass responsivity of the fundamental roof tile-shaped mode by placing silicon dioxide microbeads as test mass on defined positions across the cantilever surface and measuring the corresponding shift in resonance frequency. The results show very good agreement with finite element analyses and are used to verify an analytic expression for the position-dependent mass responsivity. This expression allows for the calculation of the mass responsivity of an arbitrary point on the cantilever surface. A mass responsivity of 24 Hz ng⁻¹ was found at the point of maximum oscillation amplitude at the free end of the cantilever, which rapidly decreases towards the nodal lines of the vibration. The 2D spatial distribution of the mass responsivity is discussed in this paper, which has to be considered in the design of high precision resonant mass sensors when targeting the roof tile-shaped mode. Since the accuracy in mass determination relies on the precise knowledge of the added mass position, the maximum error due to limited spatial accuracy is determined and the mid-section of the free end is identified to be the most suitable position on the cantilever surface for mass detection.

Keywords: MEMS, mass sensor, microcantilever, mass responsivity, resonance frequency

(Some figures may appear in colour only in the online journal)

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1. Introduction

The use of resonantly excited microelectromechanical systems (MEMS) cantilevers as mass sensors was proposed about two decades ago (Cleveland et al 1993). The working principle is based on the dependence of the cantilevers resonance frequency on the total oscillating mass. By functionalizing the surface of the cantilever it is possible to promote the adsorption of specific substances, what allows for the detection of ultralow additional mass values down to the pg-level (Johnson and Mutharasan 2012). The resulting shift of the resonance frequency can be used to deduce the accumulated mass, which makes this sensor principle useful for a wide field of applications, ranging from biochemical analysis (Buchapen et al 2011) to the detection of micro- and nano-sized particles (Wasisto et al 2013). Most studies utilize the flexural out-of-plane mode, which is well suited for mass sensing in vacuum and air. In liquids, however, the cantilever experiences high damping by the surrounding fluid what substantially deteriorates its mass sensing performance (Cox et al 2012). To circumvent this problem, higher order out-of-plane modes or different mode shapes, such as the in-plane mode, can be used for measurements in liquids (Dufour et al 2007, Van Eysden and Sader 2007). In the past, the application of the so-called roof tile-shaped modes for sensor applications was proposed (Kucera et al 2014). This special type of flexural mode has a 2D shape of vibration and shows superior performance (i.e. low damping) in liquid environments, resulting in quality factors of about 200 in water at resonance frequencies of approximately 300kHz. This class of modes is therefore well-suited for mass sensing applications in a fluid environment. Pfusterschmied et al studied the potential of piezoelectrically excited resonators for viscosity and density sensing of fluids, using the roof tile-shaped modes (Pfusterschmied et al 2015, 2017). Recently we showed the detection of fluids in particles in fluids using this class of modes, by monitoring the resonance frequency shift of a highly-integrated MEMS sensor (Patocka et al 2018). For the in-depth investigation of such mass measurements in liquid environments, a theoretical framework to connect the accumulated mass and the resonance frequency shift of the sensor is needed.

In this work, we map the spatial distribution of the mass responsivity $R = \Delta \Omega / \Delta m$ (resonance frequency shift $\Delta \Omega$ per mass change $\Delta m$) of the fundamental roof tile-shaped mode across the cantilever surface. Because we are interested in investigating the validity of extending the analytical framework of the flexural out-of-plane modes to 2D modes, we conducted these experiments in vacuum to cancel out any viscous effects. The position dependency of the mass responsivity is used to determine the mass change from the mass sensor output signal, i.e. the resonance frequency shift. An earlier study investigated the performance of the roof tile-shaped mode for immuno-sensing, but did not consider the position-dependent mass responsivity of such modes (Oliver et al 2012). Due to the 2D mode shape, the mass responsivity depends on the position of the additional mass along the width and length of the resonator. This is in contrast to the out-of-plane mode, where $R$ solely depends on the position along the cantilever axis (Dohn et al 2005). We performed these investigations by adding a small test mass in form of a spherical silicon dioxide microbead on predefined positions on the surface, thus ensuring a point-like connection, and subsequently measuring the resulting resonance frequency shift. Repeating this procedure, it is possible to map the mass responsivity across the cantilever surface. The latter measurements are evaluated against both finite element analyses (FEA) and an analytical expression, which is the extension of the analytical framework of the 1D modes to the 2D modes used in this study. Finally, the consequences for the design of resonant mass sensors, targeting the fundamental roof tile-shaped mode, are discussed. This work lays the foundation for the in-depth investigation of mass measurements in liquid environments using the class of roof tile-shaped modes of MEMS-cantilevers.

2. Materials and experimental details

Commercially available silicon dioxide microbeads with a diameter of approximately 10 $\mu$m were used as test masses. Since the mass of the used bead directly affects the measurement, it was carefully determined by first calculating the mass by measuring the size of the bead in a scanning electron microscope (see inset figure 1) and by applying the density given by the manufacturer. To validate the calculated bead mass, we used the established approach of measuring the resonance frequency shift of the fundamental out-of-plane mode in dependence of the attached mass (Dohn et al 2007). A set of six different cantilevers, ranging from 300 to 500 $\mu$m in length and from 50 to 75 $\mu$m in width, was prepared and the bead was successively transferred from one cantilever to another cantilever using a standard atomic force microscope (AFM) equipment. The frequency shift induced by the bead was measured, thus enabling to calculate and verify the previously determined bead mass $m_{\text{bead}} = 1.245 \pm 0.045$ ng.

The cantilever used for the investigation of the position-dependent mass responsivity of the fundamental roof tile-shaped mode was fabricated from a 4 inch

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$^4$ Micromod Sicar® ($\rho = 1.8 \text{ g cm}^{-3}$), http://micromod.de
The data to determine the resonance frequency at edge (E), on the nodal lines (N) and in the centre (C) where the mass is applied. The free end is located at $x = 0 \mu m$ and the cantilever is fixed at $x = 1000 \mu m$. The highlighted points show the positions along the edge (E), on the nodal lines (N) and in the centre (C) where the mass responsivity $R_m$ was measured.

The mass responsivity of a certain point on the cantilever, oscillating in the fundamental roof tile-shaped mode. The surface of the cantilever was scanned with a laser-Doppler vibrometer with a spatial resolution of approximately 13 $\mu m$. To plot the data a Gaussian filter and a cubic interpolation scheme was applied. The free end is located at $x = 0 \mu m$ and the cantilever is fixed at $x = 1000 \mu m$. The highlighted points show the positions along the edge (E), on the nodal lines (N) and in the centre (C) where the mass responsivity $R_m$ was measured.

In order to validate the measurements, FEA was conducted to calculate the eigenfrequency of the cantilever with an attached bead mass, while varying its position across the cantilever surface. The simulated resonance frequency shift was used to calculate the position-dependent mass responsivity of the FEA model $R_{FEA}$, which is given in the second row of table 1. A very good agreement with the measured values is found, whereas the largest relative deviations up to 60% are found in areas of low mass responsivity. Since the resonance frequency shift is well below 1 Hz at these positions, the limited

$$\Delta \Omega = (\Omega_{\text{Act},1} - \Omega_{\text{Act},0}) + (\Omega_{\text{Ref},0} - \Omega_{\text{Ref},1}).$$

3. Results and discussion

3.1. Measurement results and verification

The measured mass responsivity values are given in the first row of table 1. Due to limited accuracy when determining the resonance frequency, the uncertainty of these values is 0.14 Hz $\text{ng}^{-1}$. Additionally, the limited accuracy in determining the bead mass accounts for a systematic error of approximately $\pm 3.5\%$. Note that due to the symmetry of the mode shape, it is sufficient to probe only one half of the cantilever. As already demonstrated for the flexural out-of-plane mode, the mass responsivity at a certain position on the cantilever surface is closely connected with the corresponding vibrational amplitude (Yu and Li 2009). The highest mass responsivity is found in the edge regions of the free end of the cantilever, where the bead gains high kinetic energy and therefore change the resonance frequency most significantly. On the other hand, the mass responsivity close to the nodal points is negligible, as the amplitude is close to zero in these areas.

In order to validate the measurements, FEA was conducted to calculate the eigenfrequency of the cantilever with an attached bead mass, while varying its position across the cantilever surface.
Table 1. The values of the experimentally determined mass responsivity $R_{\text{Meas}}$, the simulated values $R_{\text{FEA}}$ and the values of $R_{\text{Analytic}}$ as calculated by equation (6) for the positions highlighted in figure 2. The last row contains the maximal deviation of $R_{\text{Analytic}}$ for the assumed spatial error of 2.5 $\mu$m when determining the bead position. All values are in Hz ng$^{-1}$.

|       | Centre |          |          |          |          |          | Nodal lines |          |          |          | Edge |          |          |          |
|-------|--------|----------|----------|----------|----------|----------|-------------|----------|----------|----------|------|----------|----------|----------|
|       |        | C1       | C2       | C3       | C4       | C5       | C6          | N1       | N2       | N3       | E1   | E2       | E3       | E4       | E5       |
| $R_{\text{Meas}}$ | 3.90   | 3.53     | 6.78     | 4.15     | 3.84     | 1.17     | (±0.28)     | 0.11     | 0.10     | 0.60     | 13.37| 10.31    | 7.50     | 6.10     | 1.80     |
| $R_{\text{FEA}}$  | 3.50   | 3.24     | 6.76     | 3.96     | 3.90     | 1.17     | (±0.14)     | 0.04     | 0.04     | 0.49     | 13.49| 10.10    | 6.76     | 5.79     | 1.50     |
| $R_{\text{Analytic}}$ | 3.50  | 3.21     | 6.72     | 3.93     | 3.87     | 1.18     | (±0.16)     | 0.02     | 0.02     | 0.48     | 13.37| 10.12    | 6.69     | 5.73     | 1.48     |
| $\Delta R_{\text{max}}$ | 0.11  | 0.1      | 0.02     | 0.08     | 0.04     | 0.02     | (±0.21)     | 0.38     | 0.29     | 0.21     | 0.18 | 0.18     | 0.18     | 0.06     | (All values in Hz ng$^{-1}$) |
accuracy of about 0.17 Hz when experimentally determining the resonance frequency introduces a significant error to the measurements, thus dominating the overall accuracy.

3.2. Spatial distribution of the mass responsivity

For mass sensing applications, it is desired to promote the accumulation of mass in regions with high mass responsivity. Thus, the spatial distribution of the mass responsivity is required to optimize the sensor design. The well-known formula for the resonance frequency of a cantilever, loaded with a point mass $\Delta m$ at the position $x_{\Delta m}$ along the length is generalized to the 2D case (Dohn et al 2007):

$$\Omega_{\Delta m}^2 = \Omega_0^2 \left(1 + \frac{\Delta m}{m_c} \phi^2 (x_{\Delta m}, y_{\Delta m}) \right)^{-1}.$$  

(4)

Here, $m_c$ is the mass of the cantilever, $\Omega_0$ is the resonance frequency without the point mass. Since it is assumed that the cantilever mass greatly exceeds the additional mass, a Taylor approximation for $\Delta m$ leads to the final expression for the position-dependent mass responsivity of the 2D roof tile-shaped mode

$$R_{\text{Analytic}} (x_{\Delta m}, y_{\Delta m}) = -\frac{\Omega_0 - \Omega_0}{\Delta m} = -\frac{\Omega_0}{2m_c} \phi^2 (x_{\Delta m}, y_{\Delta m}) .$$  

(5)

For the flexural out-of-plane modes, an 1D expression of the mode shape function $\phi$ can be derived analytically (Schmid et al 2016). Up to date, such an expression is not available for the 2D roof tile-shaped mode. Because of the complex mode shape, simple approximations do not yield satisfactory results. Thus, we obtained the values of the mode shape function by a FEA: an eigenfrequency study of the micro-cantilever is performed and the values of the mode shape function are extracted from these simulation results. An anisotropic material model must be used, since the orientation-dependent mechanical properties of monocrystalline silicon have a strong impact on the derived mode shape in the case of 2D modes (Hopcroft et al 2010). Since the simulated mode shape is normalized in an arbitrary manner, a suitable normalization scheme has to be applied. The mass responsivity $R_D$ of a cantilever, coated with an evenly distributed mass, is given by

$$R_D = \frac{1}{LW} \int_0^L \int_0^W R (x, y) \, dx \, dy = -\frac{1}{2m_c} \Omega_0 .$$  

(6)

with $L$ and $W$ being the length and width of the cantilever, respectively. By comparison to equation (5), it is obvious that the mode shape function $\phi(x,y)$ has to be normalized in a way that

$$\frac{1}{LW} \int_0^L \int_0^W \phi^2 (x,y) \, dx \, dy = 1 .$$  

(7)

The mode shape function, obtained by FEA, can now be used to calculate the mass responsivity at an arbitrary point $(x,y)$ on the cantilever surface. A surface plot of $R_{\text{Analytic}}$ as calculated by equation (5), is depicted in figure 3. To verify the validity of our expression, we calculated the values of $R_{\text{Analytic}}$ at the previously measured positions, which are given in the third row of table 1 and compared them to $R_{\text{Meas}}$. Excellent agreement is found, showing the validity of equation (5), which can now be used for further analysis of the position dependent mass responsivity.

Figure 3 shows that the highest mass responsivity is found at the edge regions at the free end of the cantilever with approximately 24 Hz ng$^{-1}$. As already mentioned in the introduction, our device has rather large geometrical dimensions in comparison to most resonant mass sensors found in literature, resulting in a high $m_c$ value of about 50 $\mu g$. Despite this drawback, the mass responsivity in vacuum is comparable to devices of similar dimensions (Nugaeva et al 2007, Faegh et al 2013, Wasisto et al 2015). These studies utilise the flexural out-of-plane mode, where the high viscous damping of the out-of-plane modes renders their use in a liquid environment unfeasible (Manzaneque et al 2010). In contrast, the low viscous damping of the roof tile-shaped modes allows for the
utilisation of these class of modes for mass sensing applications in liquids (Patocka et al 2018).

3.3. Accuracy of mass determination

To determine the additional mass with high precision, its position on the cantilever surface has to be known precisely. To investigate the impact of limited spatial accuracy, we calculated the maximum error for our measurements due to a limited bead position measurement of 2.5 μm. First, we determined the magnitude of the gradient of $\Delta R_{\text{max}}$ as illustrated in figure 4, and calculated the resulting maximum error $\Delta R_{\text{max}}$ for all fourteen measurements, which is given in the fourth row of table 1. For most positions, the error is well below 5%, whereas it is significantly larger for the measurements next to the nodal lines. Despite the low gradient in these areas, the error has the same magnitude as the mass responsivity, thus making these areas less attractive for mass sensing purposes. Additionally, accumulation of matter in this area can counteract the mass-induced frequency shift by ‘stiffening’ the microcantilever (Tamayo et al 2006). To maximize the overall mass responsivity of the sensor, it should be avoided to accumulate any additional mass on or even close to the nodal lines. For the fundamental out-of-plane mode this effect is only relevant close to the anchor region of the cantilever, but special care has to be taken when designing a sensor exploiting the roof tile-shaped mode, as the nodal lines spread along the whole length of the cantilever. Even though the edge of the microcantilever exhibits the largest mass responsivity (position E1), the high gradient leads to a significant error even for small deviations of the assumed mass position. The lowest error is found for position C3 being in the centre of the cantilever. Since the gradient of the mass responsivity is close to zero in this area, inaccuracies when determining the mass position have a very low impact on the measurement, whereas the error when determining the resonance frequency shift can become prominent. Provided that the resonance frequency shift is significantly larger than the uncertainty when determining the resonance frequency (e.g. 0.17 Hz for our measurements), the added mass can be determined with the highest precision in this area.

4. Conclusions

By measuring the frequency shift induced by carefully placing a microbead on the cantilever, we determined the mass responsivity of the fundamental roof tile-shaped mode at distinct positions across the surface. The measurements show very good agreement with FEA and were used to verify an analytic expression for the position-dependent mass responsivity. We found a maximum mass responsivity of 24 Hz ng$^{-1}$ at the edge regions of the cantilever. Given the large device size of $1000 \times 1000 \times 20 \, \mu m^2$, this shows the feasibility of our approach to use the fundamental roof tile-shaped mode for resonant mass sensing. Furthermore, we discussed the spatial distribution of the mass responsivity and identified the sources of errors when determining the added mass on the cantilever. Using this knowledge, the corresponding consequences for future sensor designs based on this vibrational mode were deduced. All in all, these promising results pave the way to study the mass sensing performance of higher orders of the roof tile-shaped modes even in liquid environments due to their high Q-factors.

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