MODELING, ANALYSIS, AND EXPERIMENTAL VALIDATION OF A BIFURCATION-BASED MICROSENSOR

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ABSTRACT

Microelectromechanical chemical and biological sensors have garnered significant interest over the past two decades due to their ability to selectively detect very small amounts of added mass. Today, most resonant mass sensors utilize chemomechanically-induced shifts in the linear natural frequency for detection. In this paper, an alternative, amplitude-based sensing approach, which exploits dynamic transitions across saddle-node bifurcations that exist in a microresonator’s nonlinear frequency response, is investigated. In comparison to their more traditional, linear counterparts, these bifurcation-based sensors have the ability to provide improved sensor metrics, eliminate power-consuming hardware from final sensor implementations, and operate effectively at smaller (e.g. nano) scales. The present work details the ongoing development of a bifurcation-based mass sensor founded upon the near-resonant response of piezoelectrically-actuated microcantilevers. Specifically, the work details the modeling and analysis of these devices, their functionalization, and proof-of-concept mass sensing experiments which not only validate the proposed technique, but allow for the direct evaluation of pertinent sensor metrics.

1 INTRODUCTION

Chemical and biological sensors founded upon microelectromechanical devices have garnered significant research interest over the past two decades due to their ability to selectively detect small amounts of added mass (see, for example, [1–3]). This capability, in addition to the advantages of comparatively small size, low power consumption metrics, and favorable cost, has led to the consideration of microscale sensors in applications ranging from medical diagnostics and environmental monitoring to national security and public safety [4–7]. Conventional resonant mass sensors utilize chemomechanically-induced shifts in the linear natural frequency of the system, arising from the adsorption/absorption of an analyte onto/into a selectively-functionalized surface, for mass detection. These changes in frequency can be directly correlated to an analyte detection event or can be used to determine the relative concentration of an analyte in a given test environment [8].

While there is unquestionable utility associated with sensing approach described above, recent results indicate that improved sensor metrics may be achieved by exploiting a sensor’s near-resonant, nonlinear behavior. The benefits of tracking a system’s nonlinear resonant frequency for mass detection was highlighted by Dai and collaborators [9]. Likewise, Younis, Zhang and their respective collaborators highlighted the potential benefits of mass sensors based on amplitude shifts resulting from...
FIGURE 1. FREQUENCY RESPONSE OF A DUFFING-LIKE RESONATOR WITH SOFTENING NONLINEARITY. POINTS A AND B REPRESENT SADDLE-NODE BIFURCATIONS. NOTE THAT AS THE SYSTEM TRANSITIONS FROM POINT A’ TO POINT A, THERE IS A SUDDEN JUMP IN RESPONSE AMPLITUDE (THE INSET SHOWS THE TIME RESPONSE WHEN THE SYSTEM MOVES ACROSS THE BIFURCATION POINT - IGNORING TRANSIENT DYNAMICS). THIS TRANSITION CAN BE INDUCED BY CHEMOMECHANICAL SHIFTS IN THE RESONATOR’S NATURAL FREQUENCY AND CAN BE DIRECTLY CORRELATED TO A MASS DETECTION EVENT.

operation near electrostatic pull-in and a parametric instability respectively [10, 11]. Building upon these prior endeavors, the present work seeks to develop an amplitude-based mass sensing approach that utilizes dynamic transitions across sub-critical saddle-node bifurcations that exist in the nonlinear frequency response of a prototypical microscale device.

Microscale resonators operating in a nonlinear regime typically exhibit a Duffing-like frequency response characteristic near the first natural frequency. This frequency response is characterized by the presence of multiple steady-state solutions, hysteretic behavior and two saddle-node bifurcations. Figure 1 shows the steady-state response of a representative resonator, plotted against the normalized excitation frequency (the excitation frequency normalized by the linear natural frequency of the resonator). A small change in the normalized excitation frequency of the resonator (caused by either sweeping the excitation frequency or the adsorption of an analyte onto the surface), when operating near the lower saddle-node bifurcation point causes a sudden change in the response amplitude (i.e. the system jumps from the lower amplitude solution branch to the higher amplitude solution branch). This abrupt change in the near-resonant amplitude of the resonator can be exploited for mass sensing purposes, much like a shift in the resonant frequency of the system. Note that an amplitude-based sensor offers the potential to eliminate frequency tracking hardware components, which are typically required in a linear resonant mass sensor, from final device implementations.

Piezoelectrically-actuated microcantilevers were selected as a test bed to investigate the proposed bifurcation-based sensing approach. These devices are well suited for mass sensing due to their self-sensing capabilities [12] and maturity in force sensing and in scanning probe microscopy contexts [13]. In order to fully describe the dynamics of these probes, a comprehensive nonlinear equation of motion has been developed accounting for the geometric, inertial and the inherent material nonlinearities of the composite system, as described in Section 2. Section 3 describes the analysis of the aforementioned model and includes an evaluation of the sensor’s performance metrics. Section 4 details the experimental characterization of the proposed bifurcation-based sensor, including device functionalization, frequency response characterization, and proof-of-concept mass sensing experiments. The paper then concludes with a brief summary of results and an overview of ongoing research.

2 MODELING

Figure 2 highlights, both schematically and pictorially, the piezoelectrically-actuated microbeam of interest. The microbeam consists of a silicon cantilever and an integrated ZnO layer, which is sandwiched between two Au/Ti electrodes. Figure 3 shows the beam element, with associated variables, used for modeling.

Employing classical energy techniques, the equation of motion governing the transverse vibrations of the system is derived. Defining the axial and transverse deformations of the beam as \( u(s,t) \) and \( v(s,t) \) respectively, and \( \dot{u} \) and \( \dot{v} \) as the derivative operators associated with the time and the arc length variable \( s \), the kinematic constraint relating the transverse and longitudinal deformations to the beam’s angular deflection \( \psi \) can be expressed as

\[
\tan \psi = \frac{v'}{1 + u'^2}.
\]

The axial strain associated with the microbeam \( \epsilon_{11} \) is then given by

\[
\epsilon_{11} = -(y - y_n)\psi' \quad \text{for} \quad s < l_1,
\]

\[
= -y\psi' \quad \text{for} \quad l_1 < s < l,
\]

where \( y_n \) is the location of the neutral axis from the midplane of
the silicon cantilever for the region $s < l_1$ and is given by

$$y_n = \frac{E_p w_{tp}(t_p + t_b)}{2(E_p t_p w_p + E_{db} w_b)}.$$  \hspace{1cm} (3)

Note that $E_b$ and $E_p$ are the elastic modulus of the beam and the elastic modulus of the laminate stack consisting of the Au/Ti electrodes and the piezoelectric material respectively \cite{14}, $w$ and $t$ represent the width and the thickness of different sections of the device, with the subscripts $p$ and $b$ representing the piezoelectric stack and the silicon cantilever respectively, and $l_1$, $l_2$ and $l$ are defined as in Fig. 2.

Using classical constitutive equations, the relationships between the induced stress and the strain in the silicon cantilever and the piezoelectric patch can be defined. For the silicon cantilever, the constitutive equation is given by $\sigma_{11}^{s} = E_p e_{11}^{s}$, while, for the piezoelectric patch, the constitutive relations are given by \cite{15, 16}

$$\begin{align*}
\sigma_{11}^{p} &= E_p e_{11}^{p} - h_{31} Q_3 + \frac{\alpha_1}{2} (e_{11}^{p})^2 + \frac{\alpha_2}{2} (Q_3)^2 - \alpha_3 e_{11}^{p} Q_3 \\
D_3 &= h_{31} e_{11}^{p} + \beta_{31} Q_3 + \frac{\alpha_3}{2} (e_{11}^{p})^2 + \frac{\alpha_2}{2} (Q_3)^2 - \alpha_2 e_{11}^{p} Q_3, \hspace{1cm} (4)
\end{align*}$$

where the superscripts $b$ and $p$ denote the beam and the piezoelectric layer respectively, and $\sigma_{11}$, $Q_3$, $D_3$, $h_{31}$ and $\beta_{31}$ represent the stress, applied electric field, electrical displacement, effective coupling coefficient that relates the electrical and mechanical displacements and the permittivity coefficient respectively. $\alpha_i$ ($i = 1, 2, 3, 4$) are nonlinear material coefficients. Note that an assumption that the electrical displacement is linear is made here. Also note that the electric field is the gradient of the applied potential ($\phi$) with respect to the thickness variable ($y$):

$$Q_3 = -\frac{\partial \phi}{\partial y}. \hspace{1cm} (5)$$

The absence of free charges inside the piezoelectric layer implies, $D_{3,3} = 0$ \cite{17}. This, along with Eqns. (2) and (4) leads to the conclusion that the potential must be a quadratic function in $y$. Using the aforementioned relationships along with the theory of classical elasticity, the kinetic energy $T$ of the resonator can be expressed as

$$T = \frac{1}{2} \int_{0}^{l} m(s) [\dot{u}^2 + \dot{v}^2] \, ds, \hspace{1cm} (6)$$

where $m(s)$ represents the mass density of the composite system. The potential energy $U$ associated with the system is then expressed as

$$U = \frac{1}{2} \int_{0}^{l_1} \int_{A} \sigma_{11}^{b} e_{11}^{b} \, dA \, ds + \frac{1}{2} \int_{0}^{l_1} \int_{A} \sigma_{11}^{b} e_{11}^{b} \, dA \, ds
- \frac{1}{2} \int_{0}^{l_1} \int_{A} Q_3 D_3 \, dA \, ds + \frac{1}{2} \int_{0}^{l_2} \int_{A} \sigma_{11}^{b} e_{11}^{b} \, dA \, ds
+ \frac{1}{2} \int_{l_2}^{l} \int_{A} \sigma_{11}^{b} e_{11}^{b} \, dA \, ds, \hspace{1cm} (7)$$

where $A$ represents the cross-sectional area of the composite system.

Using these as a basis for study and assuming that the beam has negligible rotational inertia, the specific Lagrangian of the given system can be defined as $\mathcal{L} = T - U$. Application of extended Hamilton’s principle results in the following variational
equation for the system:

$$
\delta H = 0 = \delta \int_{t_1}^{t_2} \int_0^L \left\{ L + \frac{1}{2} \lambda \left[ 1 - (1 + u')^2 - (v')^2 \right] \right\} \, ds \, dr \\
+ \int_{t_1}^{t_2} \int_0^L (Q_u \delta u + Q_v \delta v) \, ds \, dr,
$$

(8)

where $\lambda$ is a Lagrange multiplier introduced to maintain the inextensibility constraint and $Q_u$ and $Q_v$ are the external non-conservative forces in the longitudinal and transverse directions respectively. Note that the only external non-conservative force acting on the system is the force resulting from damping and thus, $Q_u = 0$ and $Q_v = -cv$, where $c$ is the specific viscous damping coefficient. Using third-order Taylor series approximations for $v$ and integrating Eqn. (8) successively by parts yields the equations governing the longitudinal and transverse vibrations of the system. Solving the equation governing the longitudinal vibration for the Lagrange multiplier and substituting the result in the equation for the transverse vibration, along with the expression for $u$ resulting from the inextensibility constraint, yields a single equation governing the transverse vibrations of the system. Nondimensionalizing this result by introducing a scaling of the displacement, arc length, and time variables yields a scaled, distributed parameter model for the system, which is not given explicitly here for the sake of brevity.

The complex distributed parameter model noted above can be reduced to a system of ordinary differential equations using standard modal projection techniques. Thus, an expansion for the transverse displacement of the system $\hat{v}(\hat{s}, \hat{t})$ of the form

$$
\hat{v}(\hat{s}, \hat{t}) = \sum_{i=0}^{\infty} w_i(\hat{t}) \Phi_i(\hat{s}),
$$

(9)

where $\Phi_i(\hat{s})$ is the modeshape of the individual modes and $w_i(\hat{t})$ is the displacement in the respective modes, is introduced. Since the response of the system is dominated by the first mode, the system is projected onto the first bending mode. Note that the beam geometry is discontinuous and hence the appropriate modeshape has to be formulated. To derive the modeshape of the discontinuous beam, the beam is split into three regions, with region 1 spanning from 0 to $l_1$, region 2 spanning from $l_1$ to $l_2$ and region 3 spanning from $l_2$ to $l$.

Solving the linear, unforced model for each region, the modeshape for each region can be obtained. Assembling the modeshape solutions together with appropriate boundary and continuity conditions renders the modeshape of the entire beam. Note that the modeshape is normalized here such that the amplitude at the tip of the microcantilever is equal to 1. The first and the second bending modes of a Veeco DMASP probe are shown Fig. 4.

Using the above formulation for the modeshape, projection onto the first mode of the beam yields the final, lumped-mass equation of motion for the system:

$$
\ddot{w} + \epsilon^2 \ddot{w} + (\omega_n^2 + \epsilon^2 \Delta \phi \lambda_1 + \epsilon^2 \Delta \phi^2 \gamma_1)w \\
+ \epsilon k_2 \dot{w}^2 + \epsilon^2 \Delta \phi \lambda_3 w^2 + \epsilon^2 (k_3 + \Delta \phi \lambda_3 + \Delta \phi^2 \gamma_3)w^3 \\
+ \epsilon^2 \chi (\omega_n^2 + \omega_2^2) = \epsilon^2 \eta_1 \Delta \phi.
$$

(10)

Note that $\Delta \phi = f \cos(\Omega \hat{t})$, where $f$ is the amplitude of voltage excitation and $\Omega$ is the normalized excitation frequency. The nondimensional coefficients in Eqn. (10) are strong functions of the system’s geometry, resonant modeshapes and constitutive material properties. The effective linear and the cubic stiffnesses ($\omega_n^2$, $k_3$), and the nonlinear term ($\chi$) vary strongly with system’s geometry and elastic material properties and exhibit a weak dependence on material nonlinearities, while the other terms in the expression are strong functions of the linear and nonlinear piezoelectric material coefficients. Detailed expressions for the nondimensional coefficients in Eqn. (10) are omitted here for the sake of brevity. Note that the coefficient scaling employed here was developed such that physically-relevant terms remain in a second-order perturbation analysis of the system. Also note that if linear constitutive relationships are used in place of their nonlinear counterparts, the resulting system model resembles a classical Duffing resonator.
3 ANALYSIS

To study the behavior of the system near its primary resonance, a second-order multiple scales analysis is performed. To simplify the analysis of the equation of motion, all material nonlinearities except \( \alpha_1 \) [in Eqn. (4)] are assumed to be zero (Note that ongoing experimental efforts are aimed at validating this assumption) [16]. Thus, Eqn. (10) is transformed to:

\[
\dot{w} + \omega_n^2 w + \epsilon^2 \dot{\epsilon} \dot{w} + \epsilon k_2 w^2 + \epsilon^2 [k_3 w^3 + \lambda_2 \Delta \phi w^2 + \chi (\dot{\epsilon} w^2 + \epsilon w^2)] = \epsilon^2 \eta_1 \Delta \phi. \tag{11}
\]

To facilitate the analysis, multiple time scales are introduced as follows

\[
\dot{\epsilon} = T_0 + \epsilon T_1 + \epsilon^2 T_2 + \ldots \\
\frac{d}{dt} = \frac{d}{dT_0} + \epsilon \frac{d}{dT_1} + \epsilon^2 \frac{d}{dT_2} + \ldots 
\tag{12}
\]

\[
= D_0 + \epsilon D_1 + \epsilon^2 D_2 + \ldots 
\]

Likewise, \( w(\dot{\epsilon}) \) is expanded according to

\[
w(\dot{\epsilon}; \epsilon) = w_0(T_0, T_1, T_2) + \epsilon w_1(T_0, T_1, T_2) + \epsilon^2 w_2(T_0, T_1, T_2) + \ldots \tag{13}
\]

Substituting Eqns. (12) and (13) into Eqn. (11) and equating coefficients of like powers of \( \epsilon \) yields

\[
(\epsilon^0) : D_0^2 \dot{w}_0 + \omega_n^2 \dot{w}_0 = 0, \\
(\epsilon^1) : D_0^2 \dot{w}_1 + \omega_n^2 \dot{w}_1 = -2D_0 D_1 \dot{w}_0 - k_2 \dot{w}_0^2, \\
(\epsilon^2) : D_0^2 \dot{w}_2 + \omega_n^2 \dot{w}_2 = -\dot{\epsilon} D_0 \dot{w}_0 - 2D_0 D_2 \dot{w}_0 - 2k_2 \dot{w}_0 \dot{w}_1 - k_3 \dot{w}_0^3 - \chi [w_0(D_0^2 \dot{w}_0^2 + w_2^2(D_0^2 \dot{w}_0^2))] - \lambda_2 \dot{\epsilon} \dot{w}_0 \dot{\epsilon} + \eta_1 \Delta \phi. \tag{14}
\]

Solving this set of equations using higher-order multiple scale methods, and introducing the following quantities

\[
\sigma = \frac{\Omega - \omega_n}{\epsilon^2 \omega_n}, \\
N_{\text{eff}} = \frac{9k_3 \omega_n^2 - 10k_2 \omega_n^2 - 6\chi \omega_n^4}{24}, \tag{15}
\]

where \( \sigma \) is a detuning parameter used to describe the nearness of the excitation frequency to the natural frequency and \( N_{\text{eff}} \) is an effective nonlinear coefficient, results in a relationship governing the steady-state frequency response of the system:

\[
\frac{16a_0^2 \sigma^2 \omega_n^2}{f^2 (-4 \eta_1 + a_0^2 \lambda_2)^2} + \frac{64 \left(a_0^3 N_{\text{eff}} - a_0 \sigma \omega_n^4 \right)^2}{f^2 (4 \eta_1 - 3a_0^2 \lambda_2)^2 \omega_n^4} = 1. \tag{16}
\]

FIGURE 5. FREQUENCY RESPONSE OF REPRESENTATIVE MICRORESONATOR WITH VARIOUS VALUES OF THE EFFECTIVE NONLINEAR COEFFICIENT \( (N_{\text{eff}}) \) EXCITED BY AN APPLIED EXCITATION VOLTAGE OF \( f = 10 \text{ V} \). STABLE SOLUTIONS ARE REPRESENTED USING SOLID LINES, WHILE THE UNSTABLE SOLUTIONS ARE REPRESENTED WITH DASHED LINES. NOTE THAT AS THE VALUE OF THE EFFECTIVE NONLINEAR COEFFICIENT INCREASES, THE SYSTEM SHIFTS FROM SOFTENING TO HARDENING BEHAVIOR.

Here, \( a_0 \) is the steady-state near-resonant amplitude of the system. Solving Eqn. (16) for \( a_0 \) and plotting the solutions as a function of the detuning parameter \( \sigma \) reveals the steady-state frequency response structure of the system. Note that the parameters \( \dot{\epsilon}, \omega_n, \eta_1 \) and \( \lambda_2 \) are known functions of the beam’s geometry and material properties, while the effective nonlinear coefficient \( N_{\text{eff}} \) is a function of the material nonlinearities of the piezoelectric material and hence, generally unknown.

To simulate the behavior of the system, the frequency response of the system is plotted for an excitation amplitude of \( 10 \text{ V} \) for different values of the effective nonlinear coefficient \( N_{\text{eff}} \). The output amplitude is plotted against the detuning parameter \( \sigma \), which can be directly correlated to the excitation frequency (Fig. 5). The plot shows the presence of multiple solutions, with the stable solutions being represented by the solid lines and the unstable solution being represented by the dashed lines. The values of the detuning parameter (and hence, the frequency) at which the stable and unstable solutions meet are the saddle-node bifurcation frequencies. The frequency response shows the presence of two saddle-node bifurcation frequencies, one where the lower amplitude stable solution and the unstable solution meet, and another where the higher amplitude stable solution and the unstable solution meet (not shown in figure). For negative values of the effective nonlinear coefficient, the system shows a softening behavior while for positive values, the system shows hardening frequency response characteristics.

Motivated by the above results, the excitation voltage is plot-
ted against the lower saddle-node bifurcation frequency for a given value of the effective nonlinear coefficient (Fig. 6). The plot shows that the bifurcation frequency has a near-linear relationship with the excitation voltage over the excitation range of interest. To accurately estimate the bifurcation frequency, an estimate of the effective nonlinear coefficient $N_{e,f}$ is needed. $N_{e,f}$ is a function of the material nonlinear coefficient $\alpha^t$. An estimate of the nonlinear material coefficient can be achieved by designing a simple parametric identification routine and using it in conjunction with experimental results. Illustrations of typical parametric identification routines in piezoelectrically-actuated microscale resonators can be found in [16] and [18]. Employing a similar identification scheme will provide a better estimate for the bifurcation frequency than that detailed utilized here, and will also allow for improved estimates of the sensor’s metrics.

### 3.1 SENSOR METRICS

To compare the effectiveness of a given mass sensor against other comparable platforms, it is important to define the metrics of the device both analytically and experimentally. One metric of particular interest is the minimum detectable mass or mass sensitivity of the device. For linear resonance based mass sensors, mass sensitivity is a function of the mass responsivity of the sensor and frequency resolution, and is given by [19]

$$\delta m \approx R^{-1} \delta \omega,$$

where $\delta m$ is the mass sensitivity of the sensor, $R$ is the mass responsivity of the sensor and $\delta \omega$ is the frequency resolution of the system. The mass responsivity $R$ is given by

$$R = \frac{\partial \omega_n}{\partial m_{e,f}},$$

where $\omega_n$ is the natural frequency of the system and $m_{e,f}$ is the effective mass of the system, accounting for the added mass due to the adsorption of analyte molecules onto the functionalized surface. Since $R$ is a deterministic quantity fixed by sensor selection, the mass sensitivity of a given device is commonly a strong function of the frequency resolution. In the presence of noise and uncertainty, the frequency resolution (and hence, the minimum detectable mass) is strongly governed by the minimum resolution that could be achieved from the experimental setup.

For an amplitude-shift based mass sensor, the chain rule of differentiation can be employed to derive an expression for mass sensitivity from Eqn. (17)

$$\delta m^* = R^{-1}S^{-1} \delta a,$$

where $\delta m^*$ is the mass sensitivity of an amplitude-based sensor, $R$ is the mass responsivity as defined in Eqn. (18), $S$ is the change in output amplitude as a function of the excitation frequency and $\delta a$ is the amplitude resolution, that is, the minimal detectable change in amplitude as limited by the presence of noise and uncertainty:

$$S = \frac{\partial a}{\partial \omega}.$$

Note that the sensor operates in a frequency range where multiple stable steady-state solutions are present. In the absence of noise and external perturbations, the system remains on the solution branch dictated by initial conditions until a bifurcation point is crossed, where the rate of change of amplitude as a function of the frequency tends to infinity. The minimal detectable difference in amplitude between the two stable solutions at the saddle-node bifurcation dictates the amplitude resolution of the sensor. For the given system, this quantity is quite high (as shown in Fig. 5 and later verified experimentally) and therefore, the system is not practically limited by the amplitude resolution. Thus, a bifurcation-based sensor, operating at a saddle-node bifurcation frequency in the absence of noise has a minimum detectable mass tending to zero.

The effects of noise on the bifurcation frequencies of a nonlinear system have been investigated by a number of researchers, with notable efforts emphasizing saddle-node bifurcations being
reported in [20, 21]. As shown in [20], for a typical Duffing-like resonator, noise blurs the separatrix between the basins of attraction of the two different stable solutions. Thus, when choosing the operating point for the nonlinear sensor, the excitation frequency should be selected such that noise does not cause the system to crossover into the other basin of attraction. This places constraints on the minimum detectable mass of the sensor (or, the exposure time needed for the sensor to detect the presence of analyte in the case of a threshold sensor). While a number of previous efforts have addressed the control and stabilization of the dynamic response of microresonators near bifurcations (see, for example [22]), the study of such control techniques, as well as stochastic nature of the bifurcation phenomenon, is largely beyond the scope of the present work and hence, is not addressed in detail here.

4 EXPERIMENTAL CHARACTERIZATION

To validate the operating principles of the proposed bifurcation-based sensors, a series of experimental investigations were initiated. As a first step, Veeco DMASP probes were functionalized with a polymer capable of absorbing/adsorbing the target molecules of interest. After establishing the nonlinear frequency response characteristics of a representative microcantilever, the sensor was subjected to mass sensing trials in a carefully-controlled chemical environment.

Although there exists a wide variety of methods for functionalizing microcantilevers, inkjet printing technology has gained significant research attention recently due to its ability to precisely deposit small volumes of liquid onto any planar or non-planar surface [23]. Compared to conventional fabrication techniques, inkjet technology provides high throughput, has low production cost and is relatively clean [24]. In this work, the beams were functionalized with Poly 4-Vinyl Pyridine to facilitate methanol sensing [25].

A micro-inkjet printing process with thermal actuation enabled drop formation was used to functionalize the sensors [26]. In this process, electrical current is passed through a resistor that is placed within a reservoir containing the polymer. Resistive heating causes the temperature to rise until a vapor bubble forms and subsequently collapses. This induces a wave in the fluid, which, in turn, results in the formation of a drop [27]. Each of these cantilevers were functionalized with 110 pL drops in less than 1 second. To preserve the integrity of the sensor’s surface chemistry prior to testing, the beams were stored in a nitrogen environment. A scanning electron micrograph of a representative functionalized beam is shown in Fig. 7 [28].

The experimental setup used in this work consisted of a custom-designed chemical test setup, a scanning Laser Doppler Vibrometer and associated hardware (Fig. 8). The chemical test setup was used to precisely control the chemical makeup of the environment in which the sensors were tested. The chemical test setup consisted of multiple analyte bubblers, which were connected to precision mass flow controllers and a carrier gas supply. By controlling the pressure of the carrier gas and temperature of the analyte in the bubbler and the mass flow rates in the mass flow controllers, a wide range of concentration levels were produced. The temperature of the analyte bubblers were monitored and controlled through Resistance Temperature Detectors (RTDs) and heat pads. The mixture was then diverted to an isolated test chamber which housed the sensors. The test chamber is optically-accessible from the top and has a complete temperature control similar to that of the analyte bubblers. Sealed electrical outlets are available at the base of the chamber to provide input to the sensor and to provide electrical access to the heat pads and RTDs. The exhaust from the chamber was routed to a condensing setup where the target analyte was recovered. A LabVIEW interface controlled the entire setup, providing an input to the sensor, recovering the output from the vibrometer and controlling the chemical concentration of the test chamber (which includes the control of heat pads, RTDs and the mass flow controllers).

The nonlinear frequency responses of various Veeco
FIGURE 9. FREQUENCY RESPONSE OF THE SYSTEM, AS RECOVERED VIA FORWARD AND REVERSE FREQUENCY SWEEPS, FOR AN EXCITATION VOLTAGE OF 18 Vpp AND A 160s SWEEP PERIOD.

DMASP probes were recovered using the Polytec MSA-400 Laser Doppler Vibrometer and the LabVIEW interface. After establishing the natural frequencies of a given device, frequency sweeps were performed near the first natural frequency to characterize the nonlinear frequency response behavior. Frequency sweeps were conducted at a variety of actuation voltage levels and a variety of sweep rates, ranging from quasi-static to highly non-stationary, to characterize these devices. Figure 9 shows the amplitude response of a typical Veeco DMASP probe for a sweep period of 160 seconds and an excitation voltage of 18 Vpp, a representative set of parameters which yield repeatable and predictable bifurcation behavior. The response shows the presence of saddle-node bifurcations in both the forward and reverse sweeps.

Although the analysis above clearly shows the presence of a saddle-node bifurcation frequency during a frequency sweep, a precise estimation of the jump frequency requires careful consideration of a number of factors, including (i) Noise in the response – Most microsystems are inherently noisy and, as shown by [29] for example, exhibit observable frequency response characteristics which have a strong dependence on sweep rate; and (ii) Resistive heating – The piezoelectrically-actuated sensor is prone to resistive heating which causes the material to soften, and the bifurcation frequency to decrease. Therefore, it is necessary to let the system achieve (near) thermal equilibrium before making a measurement.

4.1 MASS SENSING EXPERIMENTS

In an effort to validate the performance of the functionalized microcantilevers and the test chamber, a series of conventional, linear mass sensing trials were performed. To this end, the beam was excited with a pseudorandom signal of 2 V amplitude, a fast fourier transform of the beam’s response was recovered, and the first natural frequency was tracked as a function of time. An analyte/carrier gas mixture (Methanol/Nitrogen) was supplied for various periods of time and the system was purged periodically with nitrogen to ensure the successive adsorption and desorption of methanol molecules onto the sensor’s functionalized surface. As the methanol molecules adsorbed on the surface, the natural frequency decreased and as the molecules desorbed, the natural frequency increased, thus indicating a mass-dominated chemomechanical process [28].

To realize an amplitude-based mass sensor, an estimate of the change in bifurcation frequency as a function of the added analyte mass is required. For this trial, the beam was subjected to a series of adsorption/desorption cycles and the saddle-node bifurcation frequency was identified at each step. Figure 10 shows a representative plot of the saddle-node bifurcation frequency as a function of time, plotted in conjunction with the flow rate of the target analyte. Here, a concentration setting of 1.18% by mass of analyte in the carrier gas mixture, as specified at the mass flow controllers, was used. As evident from the figure, the bifurcation frequency decreased as the analyte adsorbed onto the surface, at a rate of 0.125 Hz/s and increased as the analyte desorbed from the surface at a rate of 0.076 Hz/s. This trend is similar to that seen with the linear natural frequency.

Motivated by the above results, an operating point was selected for each device based on that particular device’s bifurcation frequency. The operating point was typically selected near the lower saddle-node bifurcation frequency, but sufficiently far away that stochastic switching was not observed. The sensor was

then excited at constant frequency and the dynamic response was recorded. Figure 11 shows the results of a representative mass sensing trial carried out with a concentration of 0.67% by mass of the analyte in carrier gas (as specified at the mass flow controllers), with the operating frequency set at 30 Hz below the bifurcation frequency. As the analyte/carrier gas mixture was supplied to the chamber, the bifurcation frequency decreased below the operating frequency of the sensor, and the response amplitude showed a sudden jump to the higher amplitude solution branch, signaling a positive detection event. The results presented herein have been repeated with multiple devices and at various concentration levels, with very few, if any, false positives and negatives being observed to date.

5 CONCLUSIONS AND FUTURE DIRECTIONS

The results presented herein detail the modeling, analysis, experimental characterization, and ultimately demonstrate the viability of bifurcation-based resonant mass sensors based upon dynamic transitions across saddle-node bifurcations. Current efforts are focused on further characterizing the sensitivity of these devices, optimizing sensor metrics and exploiting their self-sensing nature for implementation in an integrated architecture.

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