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Bifurcation-based mass sensing using piezoelectrically-actuated microcantilevers

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In conventional implementations, resonant chemical and biological sensors exploit chemomechanically-induced frequency shifts, which occur in linear systems, for analyte detection. In this letter, an alternative sensing approach, based upon dynamic transitions across saddle-node bifurcations is investigated. This technique not only has the potential to render improved sensor metrics but also to eliminate frequency tracking components from final device implementations. The present work details proof-of-concept experiments on bifurcation-based sensing, which were conducted using selectively functionalized, piezoelectrically-actuated microcantilevers. Preliminary results reveal the proposed sensing technique to be a viable alternative to existing resonant sensing methods. © *2011 American Institute of Physics*. [doi:10.1063/1.3574920]

Microcantilever-based chemical and biological sensors offer distinct utility in a wide variety of applications, including medical diagnostics, environmental safety, and national security, due to their small size, low power consumption, high sensitivity, and comparatively low cost.[1–3] In conventional implementations, resonant sensors utilize chemomechanically-induced shifts in linear natural frequency for mass detection, which result from the adsorption/absorption of a target analyte onto/into a selectively functionalized surface layer and a corresponding change in effective mass or stiffness. This change in frequency can be directly correlated with an analyte detection event or used indirectly to determine the relative concentration of a target analyte in a test environment.[4]

Although the aforementioned linear sensing approach offers distinct utility, recent research has shown that improved sensor metrics may be achieved by exploiting nonlinear, near-resonant behaviors. For example, Dai *et al.*[5] recently highlighted the benefits of tracking a system's nonlinear resonant frequency in mass detection. Likewise, Younis, Turner, and their respective collaborators, have highlighted the benefits of sensors, which actively exploit amplitude shifts resulting from dynamic operation near an electrostatic pull-in and parametric instability, respectively.[6,7] The present letter seeks to build upon these prior endeavors through the development of a simple, bifurcation-based sensing approach, which not only has the potential to render improved sensor metrics but also to facilitate sensing in microscale and nanoscale resonators that are sensitive to ambient pressure (effective damping) levels or exhibit a limited dynamic range.[8]

Microscale resonators operating in a nonlinear regime commonly exhibit a Duffing-like frequency response characteristic near the first natural frequency, which features multiple steady-state solutions, hysteretic behavior, and two saddle-node (fold) bifurcations (Fig. 1). A change in the normalized excitation frequency of the system (the excitation frequency normalized by the linear natural frequency) caused by either sweeping the excitation frequency or a change in the natural frequency (due to the adsorption of mass on the resonator), when operating near these saddle-node points results in a sudden change in the response amplitude. The inset in Fig. 1 shows the result of a frequency sweep near the lower saddle-node bifurcation for a Duffing-like resonator with softening behavior. Here, as the system crosses the saddle-node bifurcation point, due to an increase in the normalized excitation frequency (decrease in the resonator's natural frequency), the amplitude jumps from the lower solution branch to the upper solution branch (note that while the location of saddle-node bifurcations is a function of damping, the existence of this jump behavior is largely independent of the quality factor associated with the system, provided the device is operating in air). Thus, much like a shift in resonant frequency, this abrupt change in near-resonant amplitude can be exploited for sensing purposes.

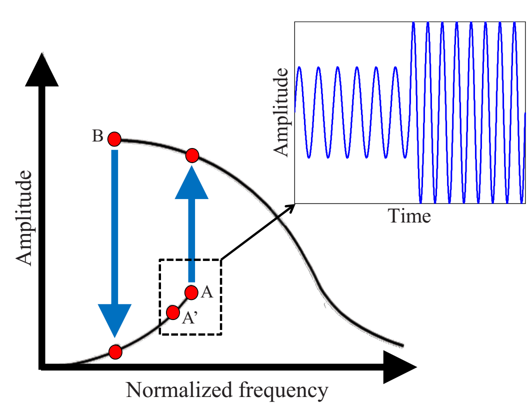

FIG. 1. (Color online) Frequency response of a Duffing-like resonator with softening nonlinearity. Points A and B represent saddle-node bifurcations. Note that as the system transitions from point A′ to point A, there is a sudden jump in response amplitude (the inset shows, schematically as transients are not highlighted, the time response when the system moves across the bifurcation point). This transition can be induced by chemomechanical shifts in the resonator's natural frequency and can be directly correlated with a mass detection event.

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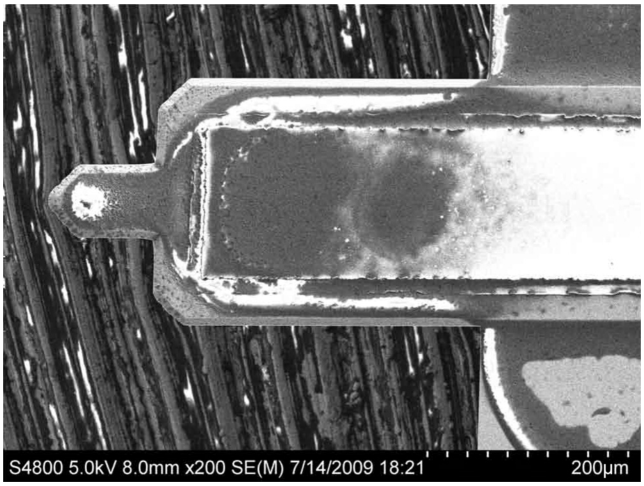

FIG. 2. SEM of a Veeco DMASP probe functionalized with poly-4 vinyl pyridine.

The present letter discusses the development of a bifurcation-based sensing approach based on amplitude shifts that occur across saddle-node bifurcations that exist in the dynamic response of piezoelectrically-actuated microcantilevers. These devices are well suited for resonant mass sensing given their inherent self-sensing capabilities[9] and maturity in force sensing and scanning probe microscopy contexts.[10]

Commercially available Veeco DMASP probes, initially designed for scanning probe microscopy applications, were selected as a test bed in this letter. These devices consist of a silicon cantilever and an integrated piezoelectric layer, incorporating a ZnO layer sandwiched between two Au/Ti electrodes. To facilitate mass sensing, the cantilevers were polymerically functionalized, using inkjet printing technology.[11] Specifically, a microinkjet printing process with thermal-actuation enabled drop formation[12] was used to functionalize the beams with poly-4 vinyl pyridine—a polymer well suited for methanol sensing.[13] Figure 2 shows a scanning electron micrograph (SEM) image of a Veeco DMASP probe post-functionalization.

The employed experimental setup consisted of a scanning laser Doppler vibrometer (SLDV) (used for optical readout in this proof-of-concept letter), a custom-designed, environmental test chamber, and associated test hardware (Fig. 3). Variations in the pressure of the carrier gas at the inlet of the bubbler, the temperature of the liquid in the bubbler, and the flow rates at the mass flow controllers were used to produce a wide range of analyte/carrier gas concentrations within the test chamber, inside of which the functionalized microcantilevers had been placed. The entire setup, including the excitation and recording of the frequency response characteristics of the beam, was controlled via a LabVIEW interface.

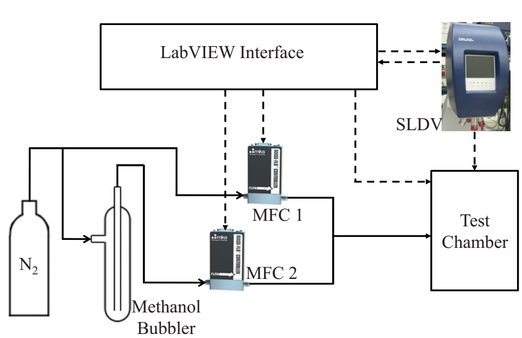

FIG. 3. (Color online) Schematic diagram of the experimental apparatus showing the chemical test setup and the SLDV.

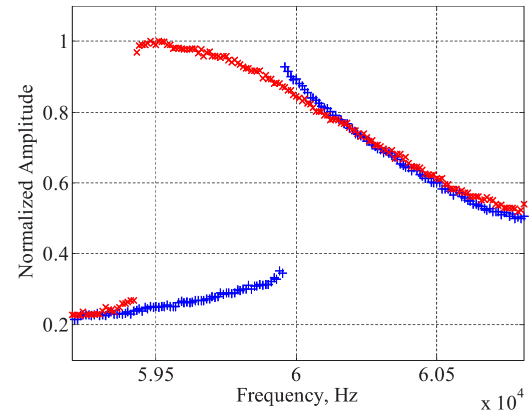

FIG. 4. (Color online) Frequency response of the system resulting from forward and reverse sweeps for an excitation voltage of 18 V_{pp} and a 1600 s sweep period.

Using the laser Doppler vibrometer, the frequency response of the functionalized beams were recovered. Frequency sweeps were performed at a variety of rates ranging from quasistatic to highly nonstationary, and at a variety of actuation voltage levels, to characterize the nonlinear behavior of the devices. Figure 4 shows the response of a representative device for a sweep period of 1600 s at 18 V_{pp}. As evident, saddle-node bifurcations are present in both the forward and reverse sweeps, along with a clear softening, hysteretic behavior. The saddle-node bifurcation frequencies were mapped as a function of the frequency sweep rates and excitation voltage levels, thus enabling the choice of an appropriate operating condition for the mass sensor.

To validate the performance of the probes and test chamber, the microcantilevers were first subjected to a series of conventional, linear mass sensing trials. Methanol/nitrogen gas mixtures were supplied for various periods of time and the system was periodically purged with nitrogen to ensure the successive adsorption and desorption of methanol molecules onto the functionalized surface. As the molecules adsorbed on the surface, the natural frequency of the devices decreased and as the molecules desorbed, the natural frequency increased, indicating a mass-dominated chemomechanical process.[14]

To study the change in bifurcation frequency as a function of the applied mass, the device was subjected to a sequence of adsorption and desorption cycles and the saddle-node bifurcation frequency during the forward sweep was identified at each step. Figure 5 shows a representative plot of the saddle-node bifurcation frequency as a function of time, plotted with the flow rate of the target analyte. This trial was performed with a concentration setting of 1.18% by mass of analyte in the carrier gas mixture (as specified at the mass flow controllers). As evident, the bifurcation frequency decreased at an approximate rate of 0.125 Hz/s as the mass was adsorbed onto the surface and increased at an approximate rate of 0.076 Hz/s as the analyte desorbed. This trend matched that previously seen with the linear natural frequency.

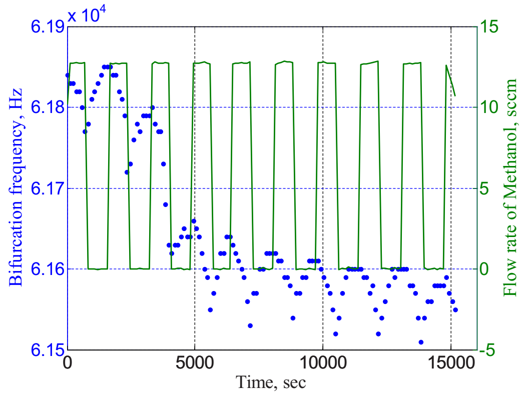

FIG. 5. (Color online) The sensor's bifurcation frequency, and the flow rate of methanol, plotted as a function of time. Note that as the mixture is supplied to the chamber the bifurcation frequency decreases. As desorption occurs, the bifurcation frequency increases.

Using the results of the aforementioned procedure as a benchmark, an operating point for each device was selected based on that particular device's bifurcation frequency. The operating point was typically a few Hertz below the saddle-node bifurcation frequency. The device was then driven at this constant frequency and the near-resonant amplitude of the device was recorded via the laser vibrometer. As the analyte/carrier gas mixture was injected into the test chamber, the bifurcation frequency decreased below the set frequency at which the system was operating and the response amplitude abruptly jumped from the low-amplitude state to the high-amplitude state, signaling a detection event. Figure 6 shows a representative plot of the response amplitude versus time for a concentration of 0.67% by mass of the analyte/carrier gas concentration (as specified at the mass flow controllers). The set frequency here was specified to be 30 Hz less than the bifurcation frequency. The positive detection event report here has been repeated with different devices and at different concentration levels, with few, if any, false positives and negatives having been observed to date.

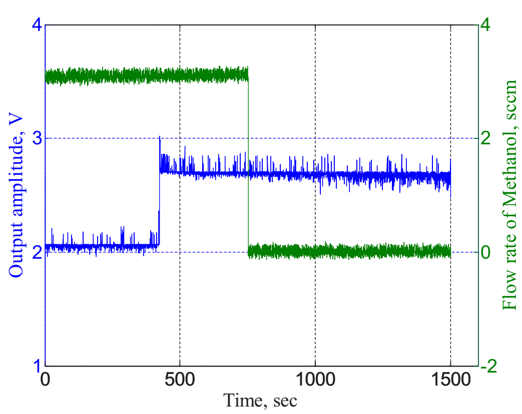

FIG. 6. (Color online) The sensor's response amplitude plotted as a function of time. As the target analyte is added, the bifurcation frequency decreases rendering a sudden jump in response amplitude. This jump in amplitude signals a detection event.

The representative experimental results highlighted in Figs. 5 and 6 clearly show that bifurcation-based mass sensors founded upon abrupt transitions in near-resonant amplitude that take place across saddle-node bifurcations are feasible. Furthermore, the letter demonstrates that with sensor calibration and exploitation of the devices' self-sensing capability, the proposed technique eliminates the need for complex frequency tracking hardware, as only amplitude monitoring or thresholding is required, and thus simplifies final device implementations. Note that to properly estimate the sensitivity of bifurcation-based sensors detailed herein, a precise estimation of the saddle-node frequency is required. The experimental determination of this frequency is limited by the stochastic nature of the switching process,[15] the rate of the frequency sweep, and resistive heating in the piezoelectric element. Current efforts are aimed at characterizing this sensitivity, optimizing sensor metrics, and exploiting the devices' inherent self-sensing capability in an integrated device architecture.

[1]T. Thundat, P. I. Oden, and R. J. Warmack, Nanoscale Microscale Thermophys. Eng. **1**, 185 (1997).
[2]K. L. Ekinci, Y. T. Yang, and M. L. Roukes, J. Appl. Phys. **95**, 2682 (2004).
[3]H. P. Lang, R. Berger, F. Battiston, J.-P. Ramseyer, E. Meyer, C. Andreoli, J. Brugger, P. Vettiger, M. Despont, T. Mezzacasa, L. Scandella, H.-J. Guntherodt, C. Gerber, and J. K. Gimzewski, Appl. Phys. A: Mater. Sci. Process. **66**, S61 (1998).
[4]T. Thundat, E. A. Wachter, S. L. Sharp, and R. J. Warmack, Appl. Phys. Lett. **66**, 1695 (1995).
[5]M. D. Dai, K. Eom, and C.-W. Kim, Appl. Phys. Lett. **95**, 203104 (2009).
[6]M. I. Younis and F. Alsaleem, J. Comput. Nonlinear Dyn. **4**, 021010 (2009).
[7]W. Zhang and K. L. Turner, Sens. Actuators, A **122**, 301 (2005).
[8]H. W. C. Postma, I. Kozinsky, A. Husain, and M. L. Roukes, Appl. Phys. Lett. **86**, 223105 (2005).
[9]M. Gurjar and N. Jalili, IEEE/ASME Trans. Mechatron. **12**, 680 (2007).
[10]K. Wolf and O. Gottlieb, J. Appl. Phys. **91**, 4701 (2002).
[11]P. Calvert, Chem. Mater. **13**, 3299 (2001).
[12]N. J. Post, Precision microdeposition of functional layers using inkjet drop-on-demand and applications to the functionalization of microcantilever sensors, M.S. thesis, Purdue University, 2007.
[13]B. E. DeMartini, J. F. Rhoads, M. A. Zielke, K. G. Owen, S. W. Shaw, and K. L. Turner, Appl. Phys. Lett. **93**, 054102 (2008).
[14]V. Kumar, J. W. Boley, H. Ekowaluyo, J. K. Miller, G. C. Marvin, G. T.-C. Chiu, and J. F. Rhoads, The 2010 SEM Annual Conference and Exposition on Experimental and Applied Mechanics, 11th International Symposium on MEMS and Nanotechnology, Indianapolis, Indiana, 2010.
[15]C. Stambaugh and H. B. Chan, Phys. Rev. B **73**, 172302 (2006).