CANTILEVER-BASED RESONANT MICROSENSOR WITH INTEGRATED TEMPERATURE MODULATION

C. Carron^{1*}, P. Getz¹, J.-J. Su¹, L.A. Beardslee^{1,2}, S.M. Heinrich³, F. Josse⁴, and O. Brand¹

¹School of Electrical and Computer Engineering, Georgia Institute of Technology, Atlanta, GA, USA

²College of Nanoscale Science and Engineering, State University of New York, Albany, NY, USA

³Department of Civil, Construction and Environmental Engineering, Marquette University, Milwaukee, WI, USA

⁴Department of Electrical and Computer Engineering, Marquette University, Milwaukee, WI, USA

ABSTRACT

This paper introduces a resonant cantilever platform with integrated temperature modulation for chemical sensing. The embedded heating elements enable rapid thermal cycling of individual resonant microstructures and, thus, allow for analysis of signal transients, which contain information about the sorption kinetics of analytes into the sensing film on the resonator surface. The signal transients are generated without the need for a microfluidic setup to switch between analyte and reference gas streams. Compared to traditional mass-sensitive microsensors operating in steady state, the on-chip generation of signal transients provides additional information for analyte discrimination and quantification.

INTRODUCTION

We live in an information-rich era that has come to expect ubiquitous on-demand data delivered at low cost to mobile platforms. This hunger for ever-increasing amounts and types of data has expanded to include chemical and biological information, with the hope that such data hold keys to improving our life. For example, MEMS-based chemical and biological sensors enable more convenient and advanced forms of Point of Care Testing (POCT), which can provide patient data in real-time at the patient's bedside [1]. MEMS-based chemical sensors, integrated with mobile platforms, further offer the possibility of distributed real-time environmental monitoring in remote or hazardous locations, where conventional techniques would be impractical or even impossible [2,3]. By adding additional value in terms of automation and real-time data collection, MEMS-based sensor technologies are even being developed to displace traditional analysis methods for addressing routine pollution concerns (e.g., municipal water supplies and indoor air quality) [2].

Currently, many standard testing systems for chemical analysis consist of bulky desktop equipment (e.g., gas chromatography, mass spectrometry), requiring costly and complex external support systems to process and treat samples with additional reagents and control fluids [2,4]. Such systems are often expensive and difficult to transport, requiring them to be located at a fixed position in a centrally located facility. Thus, many such standard tests require samples to be transported off-site to a designated testing facility for processing and analysis, which introduces significant time delays and can even lead to inaccuracies in the results due to sample degradation [2]. In instances where the sample analysis leads to a time-sensitive decision (e.g., clinical decision, quarantine of a contaminated location), such delays severely limit the efficacy of the information being gathered. In these cases, the real-time nature of data provided by low-cost MEMS-based sensors can prove more valuable than the superior accuracy of traditional techniques, leading to applications where 'good enough,' in terms of measurement resolution, can be a preferable alternative.

With the goal of developing such real-time mobile sensing platforms, this work demonstrates a resonant cantilever platform with integrated temperature modulation for chemical sensing. The

embedded heating elements enable rapid thermal cycling of individual resonant microstructures and, thus, allow for analysis of signal transients, which contain information about the real-time sorption kinetics of analytes into the sensing film on the resonator surface [5,6].

Figure 1: SEM micrograph of resonant microstructure with semicircular head with 200 μm outer radius supported by 75 μm wide and 100 μm long cantilever. Resistors for thermal excitation and piezoresistive detection of in-plane vibrations are located at the cantilever base. Three heating resistors, for rapid thermal modulation, connected in series are clearly visible on the semicircular head.

By generating the signal transients through temperature modulation with integrated heating units, this mode of operation potentially eliminates the need for an external microfluidic setup to switch between analyte and reference gas streams. Additionally, the rapid temperature modulation enables intrinsic drift compensation without the need for a reference resonator and generates measurement data in real-time. Combining these two advantages – no need for external flow system or reference resonator – can dramatically reduce the complexity and cost of the total system, leading to increased deployability in the field as part of low-power mobile platforms. Finally, when compared to traditional mass-sensitive microsensors operating in steady state, the on-chip generation of signal transients provides additional information for analyte discrimination and quantification, which can lead to improved sensing performance in the presence of interfering compounds [5,6,7,8].

DESIGN & FABRICATION

Figure 1 shows an SEM micrograph of a fabricated microresonator consisting of a semicircular head region with inner and outer diameters of 100 μm and 200 μm , respectively, supported by a 75 μm wide and 100 μm long cantilever beam. Three integrated heating resistors, connected in series, are defined around the perimeter of the semicircular head region and were formed via high-temperature boron diffusion through an oxide mask. As the thermal

excitation resistors as well as the piezoresistive sensing resistors connected in a Wheatstone bridge are formed in like manner, the integration of the embedded heating resistors does not require any additional process steps. The total series resistance of the embedded heating unit was designed to have a value of approximately 1 k Ω , allowing for significant temperature elevation with modest applied heating current in the low milliamp range. The overall resonator structure was further designed to allow for rapid and uniform heating of the head area with a minimal thermal time constant.

Figure 2: Thermal-modal analysis of resonant microstructure using COMSOL. The applied heating power of 100 mW causes a 71°C temperature elevation of the head structure (see color coding). The simulated in-plane resonant frequency of the heated structure is 776.19 kHz, representing a shift of -610Hz compared to the unheated structure.

The fabrication approach for this device represents a slight departure from our previous work [9,10,11], in that the sensing platform is formed on silicon-on-insulator (SOI) substrates and does not utilize KOH etching to release the finished devices. Instead, a high-aspect-ratio deep reactive ion etching (DRIE) process is implemented on the reverse-side of the wafer, with the buried oxide (BOX) layer serving as the etch stop. The micromachining used to pattern the top-side of the wafer is similar to our previous designs, with the exception that the DRIE used to pattern the resonator geometry from the top side does not accomplish a final release of the devices but, as with the reverse-side DRIE, stops at the BOX layer. Final release is performed by removing the exposed BOX layer regions with a standard plasma etch for SiO₂. While this process arrangement introduces additional complexity into the fabrication of the resonant devices when compared to our previous work, it also offers the ability to define high-aspect-ratio features onto the reverse-side of the die, making possible novel future designs.

Device operation remains similar to our previous work [9,10,11]. The resonator is driven at its fundamental in-plane resonance mode using the aforementioned diffused silicon excitation resistors at the base of the cantilever beam. Four piezoresistors arranged in a U-shaped Wheatstone bridge configuration generate a sensing signal at the desired in-plane mode and enable closed-loop operation of the resonator at its in-plane resonance frequency of approximately 780 kHz. Typical Q-factors for these devices are approximately 2000 in air.

SIMULATION & CHARACTERIZATION

Prior to fabrication, the device performance was simulated and the device geometry was optimized *via* Finite Element Analysis (FEA). For a 25 μ m thick resonator, finite element simulations indicate a maximum temperature elevation of 71°C at 100 mW of applied heating power and a uniform temperature profile across the heated head area of the cantilever (Figure 2). This simulated result compares well with experiment: for an applied heating power of 100 mW, the observed temperature increase of the device was ~85°C in air. This result was obtained by placing the device in a temperature-controlled chamber while continuously monitoring the change in resistance of the three series-connected heating resistors as a function of the chamber temperature, and subsequently comparing these data to the change in resistance as a function of applied heating power due to self-heating of the embedded heaters. The discrepancy between the simulated and observed temperature elevations is approximately 15%, and can be explained by taking into account differences between the simulated model and the physical device (e.g., thickness variations). Figure 3 shows the open-loop amplitude transfer characteristic of the fundamental in-plane mode as a function of the applied heating voltage. The resulting frequency drop is linear with the heating power with a slope of -10.1 Hz/mW, or -13 ppm/mW.

Figure 3: Amplitude transfer characteristic of in-plane resonant mode as a function of the applied heating power. The insert shows the in-plane resonant frequency as a function of the applied heating power with a slope of -10.1Hz/mW or -13ppm/mW.

The thermal time constant of the heated resonator can also be estimated using finite element modeling, and was found to be approximately 1.2 ms for a 25 μ m thick resonator. By applying a square-wave heating pulse with a frequency of 100 Hz to the three series-connected heating resistors and measuring the temperature-dependent resistance change of the Wheatstone bridge, a thermal time constant of 1.1 ms was experimentally observed, confirming the simulated results (Figure 4). It is important to note that this thermal time constant is 2-3 orders of magnitude shorter than typical analyte diffusion times into polymeric sensing films with micrometer thicknesses [10]. As a result, the resonators can be quickly heated to desorb the analyte out of the polymer film and then rapidly cooled to monitor the analyte absorption back into the polymer in real time. As a result of the rapid cooling of the device, the resonator quickly returns to its original temperature (i.e. the

baseline temperature of the device, prior to generating the self-heating transient) and analyte absorption into the polymer sorbent film mostly takes place at room temperature. Because the analyte sorption is recorded at the original operating temperature, the unavoidable frequency shift associated with heating the resonator does not affect the measurement.

Figure 4: Screen capture of oscilloscope showing the square-wave signal applied to the three series heating resistors on the head region of the resonator, and the voltage across the piezoresistors at a constant current of 1 mA. The extracted thermal time constant is 1.1 ms.

CHEMICAL MEASUREMENTS

Following fabrication, and subsequent electrical and thermal characterization, several micro-resonators were coated with sorbent polymer layers and evaluated for performance as gas-phase chemical sensors. Building on our previous work [11], sorbent polymer layers were applied *via* localized polymer deposition through a shadow mask onto the semicircular head region only. This way, the quality factor is preserved, even in the case of relatively thick sorbent layer films, since the head deformation is negligible; thus, the overall chemical sensitivity can be improved. The coated resonators were then embedded into an amplifying feedback loop and exposed to defined concentrations of volatile organic compounds (VOCs) in a custom gas-flow setup. The gas setup is implemented with high-precision mass flow controllers (MFCs) for flowing precise concentrations of analyte-loaded and reference carrier gas streams over the devices at known flow rates and at known temperatures.

Figure 5a summarizes a typical chemical measurement performed with a 25 μm thick heated resonator coated with a 2 μm thick polyisobutylene (PIB) sorbent polymer layer. After exposing the microstructure to pure carrier gas (N_2), it was subjected to a continuous flow (at 80 ml/min) of toluene at a concentration of 6800 ppm, at a constant temperature of 20°C. The resulting initial frequency drop of ~ 1.2 kHz is due to absorption of toluene into the PIB sensing film. While exposing the sensor to a constant toluene concentration, heating pulses of varying power were applied to the three series-connected resistors located on the head region of the resonator, and the resonance frequency of the device was continuously monitored and recorded. As can be seen in Figure 5a, the resonance frequency drops quickly when heating power is initially applied due to a rapid increase in the device temperature, which reduces the stiffness of the cantilever beam (due to the temperature dependence of the Young's modulus). Almost immediately after the heating pulse is applied, the frequency slowly begins to increase as analyte desorbs from the heated polymer film. After the heating power is turned off, the frequency rapidly shifts

upward due to the temperature decrease as device and polymer return to their initial state (as demonstrated before, the thermal time constant is approx. 1.1 ms) and then slowly decreases again as analyte is re-absorbed into the polymer layer.

The observed sorption time constant is specific to the particular (or a particular class of) polymer-analyte pair and can be used to improve analyte discrimination, as illustrated in Figure 5b. This plot of normalized frequency change vs. square-root of time compares transients induced by modulation of the integrated heating resistors with a conventional transient induced by rapidly switching between analyte-loaded gas and reference gas streams with a mechanical valve.

Figure 5: (a) In-plane resonance frequency of 25 μm thick microstructure subject to various levels of applied heating power; the PIB-coated resonator is exposed initially to pure carrier gas (N_2) and then to a constant toluene concentration of 6800 ppm; Blue lines represent analyte absorption phases, red lines analyte desorption phases; (b) Normalized frequency change vs. square-root of time for absorption transients induced by on-chip heating/cooling ($P = 40$ mW, red symbols) and analyte switching using a gas set-up (black symbols); the slope of the linear portion (solid line) is proportional to $\sqrt{D}$ and yields a diffusion coefficient, $D = 2.4 \times 10^{-9} \text{ cm}^2/\text{s}$ [10]. The heating-induced transient agrees very well with the gas-switching-enabled transient.

By plotting the ratio of the frequency change to the maximum frequency change versus the square-root of time, the initial slopes of the transients, which are proportional to the square-root of the diffusion coefficient, D [10], are observed to agree within 5%. Thus, signal transients generated *via* modulation of the on-chip heating units are nearly indistinguishable from those generated due to sudden changes in analyte concentration (e.g., switching *via* mechanical valving between analyte and pure carrier gas streams). As such, heater-generated transients carry virtually identical information as the conventional transients and can be used in like manner for chemical sensing of VOCs in the gas-phase. The extracted diffusion coefficient for toluene into PIB is $2.4 \times 10^{-9} \text{ cm}^2/\text{s}$.

Since the transients are generated in a constant-concentration environment, operation in this mode does not require an external flow system for switching between analyte and reference gas streams, thereby reducing cost and complexity in the total sensing platform.

As another example, Figure 6 represents a series of measurements performed with a resonator coated with an 11 μm thick PIB film exposed to varying concentrations of toluene. The measurements were each performed using the previously-mentioned gas-flow setup, with a constant flow rate of 80 ml/min and at a constant temperature of 20°C. In each case, 18.8 mW of heating power were applied to the device for a duration of 30 sec once the resonator became saturated with the given toluene concentration. As can be seen from the figure, the slopes of the transients agree very closely and are independent of the analyte concentration for relatively low analyte concentration levels.

Figure 6: Normalized frequency change vs. square-root of time for the initial portion of the absorption transients induced by on-chip heating/cooling ($P_{\text{applied}} = 18.8$ mW) for resonator coated with an 11 μm thick PIB film exposed to varying concentrations of toluene; the slope of the linear portion is proportional to $\sqrt{D}$ and independent of concentration, for low concentration levels.

Analysis of the measured frequency shifts reveals that the sensors exhibit a linear response with respect to toluene concentration. Another important observation from the data is that the sensor's response is fully reversible and the measurements are made in real-time due to the rapid generation of heating transients. From the baseline frequency data, a short-term frequency stability of 2×10^{-7} was extracted via the Allan variance method. Using an experimentally determined chemical sensitivity of 0.18 Hz/ppm for toluene and the Allan variance of 2×10^{-7} , a limit of detection (LOD) of approximately 3 ppm can be expected for this device. However, this LOD value was deduced via extrapolation from much higher measured concentrations and must be confirmed by measurements at actual low-ppm level analyte concentrations.

CONCLUSION

The design, fabrication, and characterization of a novel chemical sensor platform with integrated heating units for the on-chip generation of real-time heating transients is demonstrated. The heater-generated transients are shown to carry nearly identical information as compared to conventionally-generated transients (e.g., due to an abrupt change in analyte concentration), enabling a novel mode of sensor operation. This novel operation mode eliminates the need for a reference resonator and external flow system, with a corresponding reduction in total system cost and complexity. Driving down the complexity of chemical sensing systems is essential to improving system deployability in the field

and addressing the growing need for real-time information. Furthermore, the thermally-generated signal transients provide additional information for analyte discrimination and quantification. Future work will explore improving the LOD of these devices and the possibility of operating arrays of individually-coated sensors with the goal of reducing the platform's sensitivity to chemical interference.

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CONTACT

*C. Carron, Tel: +1-404-997-8623; ccarron3@gatech.edu